Salt forms of sulfadiazine with alkali metal and organic cations.

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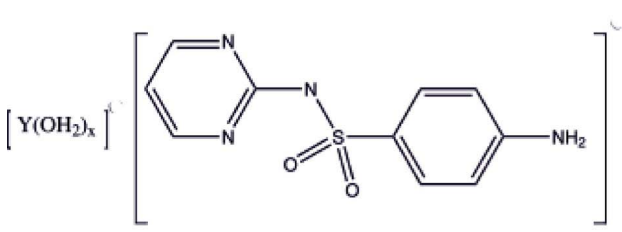
Abstract

The structures of four salt forms of sulfadiazine (SDH) with alkali metal cations are presented. Three contain the deprotonated SD anion. These are the discrete complex $[Li(SD)(OH_2)_2]$, (I), and the coordination polymers $[Na(SD)]_n$, (II), and $[K(SD)(OH_2)_2]_n$, (III). The Na complex (II) is a three-dimensional coordination polymer whilst the K complex (III) has two crystallographically independent $[K(SD)(OH_2)_2]$ units per asymmetric unit, $Z' = 2$, and gives a two-dimensional coordination polymer whose layers propagate parallel to the crystallographic *ab* plane. The different bonding modes of the SD anion in these three complexes is discussed. Structure (IV) contains protonated SDH_2 cations and the Orange G (OG), $C_{16}H_{10}N_2O_7S_2$, dianion in a structure with formula $[SDH_2]_2[Na(OG)(OH_2)_4]_2{\cdot}3H_2O$. The $[Na(OG)(OH_2)_4]_2$ dimers have antiparallel naphthol ring structures joined through two Na centres that bond to the hydrazone anions through the O atoms of the ketone and sulfonate substituents. The structures of the salts formed on reaction of SDH with 2-aminopyridine and ethanolamine are also presented as $[C_5H_7N_2][SD]$, (V), and $[HOCH_2CH_2NH_3][SD]{\cdot}H_2O$, (VI), respectively. Structure (V) features a heterodimeric $R_2^2(8)$ hydrogen bond motif between the cation and the anion whilst structure (VI) has a tetrameric core of two cations linked by a central $R_2^2(10)$ hydrogen bonded motif which supports two anions linked to this core by $R_3^3(8)$ motifs.

Keywords: crystal structure, active pharmaceutical ingredients, salt selection, sulfadiazine, sulfa drugs

53scheme1.tif



(I) Y = Li, x = 2

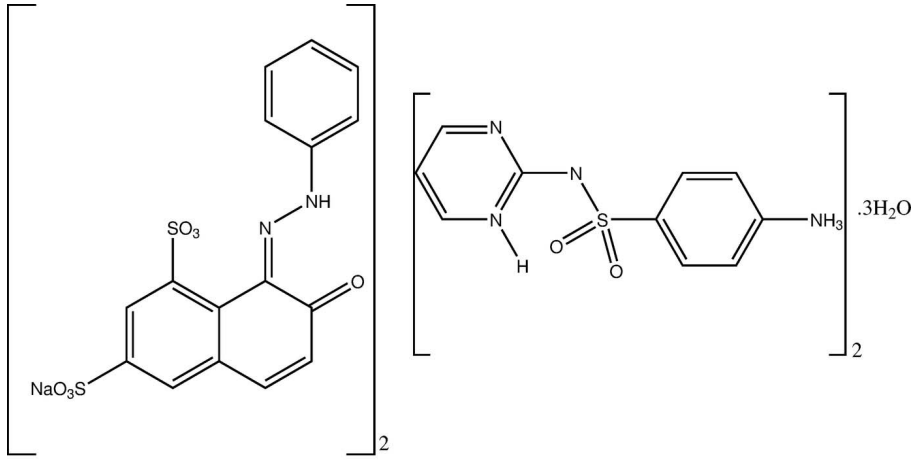
(II) Y = Na, x = 0

(III) Y = K, x = 2

(V) Y = $C_5H_7N_2$, x = 0

(VI) Y = $HOC_2H_4NH_3$, x = 1

53scheme2.tif



(IV)

1. Comment

The active pharmaceutical ingredient (API) 4-amino-*N*-(pyrimidin-2-yl)-benzenesulfonamide is commonly known as sulfadiazine (SDH) and is a well known antibiotic. Common variants are its Ag(I) complex which is used in creams and impregnated medical devices, and its Na salt which is used intravenously (Fisher *et al.*, 2003; Ghedini *et al.*, 2017; Mohseni *et al.*, 2016; Preskey & Kayes, 1976). SDH is amphoteric, allowing salt formation reactions to be carried out with both acids and bases. This is a pharmaceutically useful as salt formation is the commonest route used to modify the performance critical material properties (*e.g.* aqueous solubility, melting point or mechanical properties) of APIs (Stahl & Wermuth, 2008).

Structural studies of protonated SDH_2 cations as a variety of salt forms have been published, (*e.g.* Pan *et al.*, 2013; Buist *et al.*, 2014) as have studies of cocrystal phases featuring neutral SDH (*e.g.* Elacqua *et al.*, 2013). Structures of the deprotonated SD anion are also well represented in the literature. These include the structure of the commercially utilized Ag(I) complex (Baenziger & Struss, 1976) and of many transition metal complexes, especially those involving the heavier first row transition metals Co, Ni, Cu and Zn (*e.g.* Shi *et al.*, 2015; Sun *et al.*, 2016; Pan *et al.*, 2012). Salt structures of SD with organic cations are also well known (*e.g.* Elacqua *et al.*, 2013; Heren *et al.*, 2006). Somewhat strangely, there are very few structural studies of s-block metal complexes of SD. As far as we are aware, the only known s-block metal structure is that of a Ca salt form (Tommasino *et al.*, 2011). Given the ubiquity of s-block metal salt usage in pharmaceutical materials in general, and the long-standing commercial use of [Na][SD] in particular, this seemed an odd omission (Stahl & Wermuth, 2008; Preskey & Kayes, 1976). The current study adds to our knowledge of sulfadiazine structural chemistry by reporting the structures of three alkali metal salt forms of SD with Li, Na and K, structures (I), (II) and (III) respectively, as well as the structure formed when the sulfonated azo dye sodium Orange G (OG) crystallizes in the presence of SDH to give a form containing both Na and SDH_2 cations, (IV). Finally, the structures of two new organic salt forms (V) and (VI), prepared by reaction of SDH with the bases 2-aminopyridine and ethanolamine, are also presented for comparison.

The structure of the lithium salt (I) was found to consist of a simple discrete coordination compound of type [Li(SD)$(OH_2)_2$], see Figure 1. Extensive hydrogen bonding creates a three-dimensional hydrogen bonding network, Table 3. The SD anion acts in a chelating fashion to form a 6-membered [LiOSNCN] ring through Li to O and Li to heterocyclic N bonds. Thus although the Li coordination geometry is tetrahedral, the bond angles are considerably distorted due to the small bite angle of the chelate (range 90.91 (18) to 126.9 (2) °), see Table 2 for details of bond lengths and angles. This O,*N* chelation mode is unusual. Discrete d-block metal complexes of SD normally bond to metal centres through a N,*N* chelate utilizing both the ring and sulfamide N atoms (*e.g.* Shi *et al.*, 2015; Sun *et al.*, 2016; Pan *et al.*, 2012). The same is true of the Ca salt (Tommasino *et al.*, 2011). The polymer $[Zn(SD)]_n$ does feature a similar O,*N* chelation mode (Yuan *et al.*, 2001) and the dimeric species $[Cu_2(SD)_4]$ can be described as containing Cu—O bonds, but these are very long (2.55 to 2.75 Å) and form 4-membered chelate rings through interactions with the sulfamide N atom rather than the 6-membered ring involving the aromatic ring observed for (I) (Shi *et al.*, 2015). The structure of the pharmaceutically important coordination polymer $[Ag(SD)]_n$ does contain the O,*N* chelation mode seen for (I) with the polymer propagating through both the common N,*N* chelation mode and the O,*N* mode (Baenziger & Struss, 1976).

Like the Ag complex, the structure of anhydrous $[Na(SD)]_n$ (II) forms a coordination polymer through each SD anion making two chelating interactions with two metal centres. One such interaction forms a 6-membered NaOSNCN ring with a O,*N* bonding mode and the other forms a NaNCN ring with a N,*N* mode. The coordination polymer is further connected by NaONaO 4-membered rings, see Figs 2 and 3. Thus the atoms N1, N2, N3 and O1 bond to Na centres and form one-dimensional coordination chains that propagate parallel to the crystallographic *a* direction. Unlike the Ag

complex, in (II) the amine group of the ligand also takes part in bonding. This gives 6 coordinate Na centres, Table 4. This last interaction type links the individual chains through amine to Na bonds and gives the overall three-dimensional coordination polymer shown in Fig. 4. We are aware no other examples of anionic SD bonding to metal through its NH_2 tail. The coordination polymer is supported by amine to SO_2 N=H···O hydrogen bonds as described in Table 5.

The K salt (III) is a dihydrate with two crystallographically independent $[K(SD)(OH_2)_2]$ units per asymmetric unit, $Z' = 2$, see Fig. 5. Of the 4 independent water molecules, two (O2W and O3W) act as terminal ligands whilst O1W bridges between K1 and K1′ (′ = $1 - x$, $1 - y$, $1 - z$) and O3W interestingly bridges between three K centres (K1, K2 and K1″ where ″ = $-x$, $1 - y$, $1 - z$), see Table 6 for details. The two independent SD anions also have different bonding modes, the ligand containing O3 utilizes both of its O atoms and 3 of its N atoms to bond between 4 separate K centres. Each N atom bonds to only one K centre with the O atoms both bridging between two K centres. Like the Na and Ag species, above, this ligand features both N,*N* 4-membered ring forming and O,*N* 6-membered ring forming chelation modes. Perhaps surprisingly, the other SD anion adopts a bonding mode that forms only two donor to K contacts and bonds to only one K centre. Atoms O1 and N1 (the sulfamide N atom) bond to K2. This O,*N* [KOSN] 4-membered ring forming bonding mode is not seen for other s-block metals with SD but can be observed in the structure of $[Cu_2(SD)_4]$ (Shi *et al.*, 2015). See Table 12 for a summary of the different bonding modes adopted by SD with alkali metals. A final difference between the two SD ligands is conformational. The SD anion that bonds to multiple K centres has the same conformation as that found for the SD anions in all the other structures reported herin. That is, one of the SO_2 O atoms lies close to the plane of the aniline ring and the pyrimidine ring is *syn* to this O atom. However, the SD anion that makes only 2 bonds to K2 has an alternative conformation where it is the amide N atom and not an O atom that lies closest to the plane of the aniline ring (N1S1C5C6 = 17.7 (4) °). The coordination bonds combine to give two-dimensional coordination polymers that propagate parallel to the *ab* plane and gives the layered structure shown in Fig. 6, with organic bilayers and inorganic layers alternating along the crystallographic *c* direction. Hydrogen bonds from the amine group link between neighbouring two-dimensional coordination polymers, Table 7.

Crystallized from SDH in the presence of acid and the sodium salt of OG, (IV) has the general formula $[SDH_2]_2[Na(OG)(OH_2)_4]_2{\cdot}3H_2O$, Fig. 7. We are unaware of any other structure containing both a metal cation and cationic SDH_2. There are 4 independent water ligands bound to Na1. These are ordered but the non-coordinated water molecules are not and have been modelled over 3 crystallographic sites, each with site occupancy factor of 0.5. In common with other sulfonated azo species based on naphthol units, the dye adopts the hydrazone tautomeric form with protonation at N6 of the N=N group (Kennedy *et al.*, 2012). This leads to characteristic lengthening (*e.g.* N=N, C—C) and shortening (*e.g.* O—C, N—C) of relevant bonds compared to similar azo species. Compare values in Table 8 with *e.g.* 1.253 (2) and 1.418 (3) Å for N=N and N—C bond lengths in a typical azo species (Kennedy *et al.*, 2001). The dimeric unit is shown in Fig. 8 and is crystallographically centrosymmetric, $Z' = 0.5$. The naphthol units lie antiparallel to each other with an octahedral Na centre at each end. In addition to the 4 terminal water ligands, each Na centre bonds to the ketone group of one anion and to atom O7 of a sulfonate group of a second anion thus creating the dimeric unit. See Tables 8 and 9 for bonding parameters. There is some degree of π to π stacking across this dimer as is indicated by a minimum C···C distance of 3.482 (2) Å (between C11 and the C15 atom at $-x$, $1 - y$, $-z$). Orange G is exceptional amongst sulfonated azo colourants in having being widely studied crystallographically. Structures are known for its s-block metal salts, for its complexes with transition metals and for salt forms with organic cations (Kennedy *et al.*, 2006; Ojala *et al.*, 1994*b*; Kennedy *et al.*, 2010; Ojala *et al.*, 1994*a*). However, the dimeric unit observed here is not seen for other s-block metal salt forms of OG. The closest motif occurs in the Ag(I) complex of OG, where a similar dimer forms part of a larger polymeric coordination network (Kennedy, *et al.*, 2006). The sulfadiazine cation is found to have three H atoms bound to N4,the aniline group, and one H atom bound to N3 of the pyrimidine ring. In contrast, previous work had found that

SDH_2 cations adopted this tautomeric form with simple anions but with sulfonate anions gave the alternative tautomer with protonation at the amide N atom (Buist *et al.*, 2014). The geometry of the SDH_2 cation is somewhat different from those of the SD anions in the other structures presented. Conformationally, it is again an O atom that lies closest to the plane of the aniline ring, but in contrast to the SD anions this O atom is anti to the pyrimidine ring. An expected small increase in S—N bond length is also seen for SDH_2 as compared to the SD anions (Elacqua *et al.*, 2013; Buist *et al.*, 2014). Another feature common in other structures with SDH_2 cations is a centrosymmetric $R_2^2(8)$ hydrogen bonded dimer of cations formed utilizing amide and pyrimidine N atoms (Buist *et al.*, 2014). This motif is retained in (IV). Structure (IV) has a layered structure with hydrophylic/inorganic layers parallel to the *bc* plane alternating with hydrophobic/organic layers.

Formed from the reaction of SDH with 2-aminopyridine, structure (V) is that of $[C_6H_4N(H)NH_2][SD]$, Fig 9. It features a heterodimeric $R_2^2(8)$ hydrogen bond between the cation and the amide N atom and one pyrimidine ring N atom of the SD anion, Fig 10. There are also anion to anion interactions with the amine group of the SD anion donating two hydrogen bonds to O atoms of two neighbouring SD anions. A final hydrogen bond is of type N—H···N and is donated by the pyridine amine group to the SD amine group, see Table 10 for details. Unusually this leaves one N atom of the pyrimidine ring with no hydrogen bonding interaction. The resulting structure has layers formed of heteroaromatic rings (both pyridine and pyrimidine) alternating with layers of aniline groups, these layers lie parallel to the crystallographic *ac* plane.

Structure (VI) was obtained on the reaction of SDH with ethanolamine and is that of the hydrate, $[HOCH_2CH_2NH_3][SD]\cdot H_2O$, Fig 11. The dimeric $R_2^2(8)$ hydrogen bonded motif does not occur in this structure. It is replaced by a tertrameric unit comprising the amide N atom and one pyrimidine ring N atom of two SD anions interacting with a centrosymmetric dimer of $HOCH_2CH_2NH_3$ cations, see Fig 12. Thus there are two $R_3^3(8)$ motifs supported by a central $R_2^2(10)$ motif. The tetramers are linked by hydrogen bonding through a bifurcated bond from H4N to O and N acceptors on a SD anion and through the water molecules. Each water molecule interacts with 4 neighbouring SD anions. Thus each water molecule donates two hydrogen bonds to O atoms of SD anions and accepting two hydrogen bonds from the SD amine groups, see Table 11 for details.

2. Synthesis and crystallization

The simple salt forms were prepared by reacting 1:1 molar mixtures of sulfadiazine and MOH (M = Li, Na, K) or the organic base in 50:50 water:ethanol. The mixtures were stirred and heated to give clear solutions before being left to cool to room temperature. Partial evaporation of these reaction mixtures over 4 to 7 days gave suitable crystals of (I), (III), (V) and (VI) but a fine powder for the Na salt. Good quality crystals of the Na salt (II) were obtained by vapour diffusion of ethanol into an aqueous solution of sodium sulfadiazine. The Na OG complex (IV) was obtained by dissolving 0.20 g, 0.44 mmol of NaOG in the minimum amount of water. A slight excess of sulfadiazine (0.12 g, 0.48 mmol) was also dissolved in the minimum amount of water. The two solutions were mixed together with stirring and acidified with concentrated HCl. After 3 days, orange crystals of (IV) had grown.

3. Refinement

For all structures, H atoms bound to C atoms were placed in the expected geometric positions and treated in riding modes with $U(H)_{iso} = 1.2U(C)_{eq}$. For aromatic groups C–H = 0.95 Å and for CH_2 groups C–H = 0.95 Å. All H atoms bound to N were refined freely and isotropically as were H atoms bound to O in structure (VI). H atoms of water molecules in (I), (III) and (IV) were located by difference syntheses and required restraints to be applied such that O—H = 0.88 (1) Å and H···H = 1.33 (2) Å. For these atoms of (III) $U(H)_{iso} = 1.5U(O)_{eq}$, whilst for (I) and (IV) $U(H)_{iso}$ were refined.

For (IV), after several trial calculations, the three non-coordinated water molecules were given site occupancy factors of 0.5. For (III) the H atoms of one water ligand were modelled as disordered over three sites.

fig1.tif

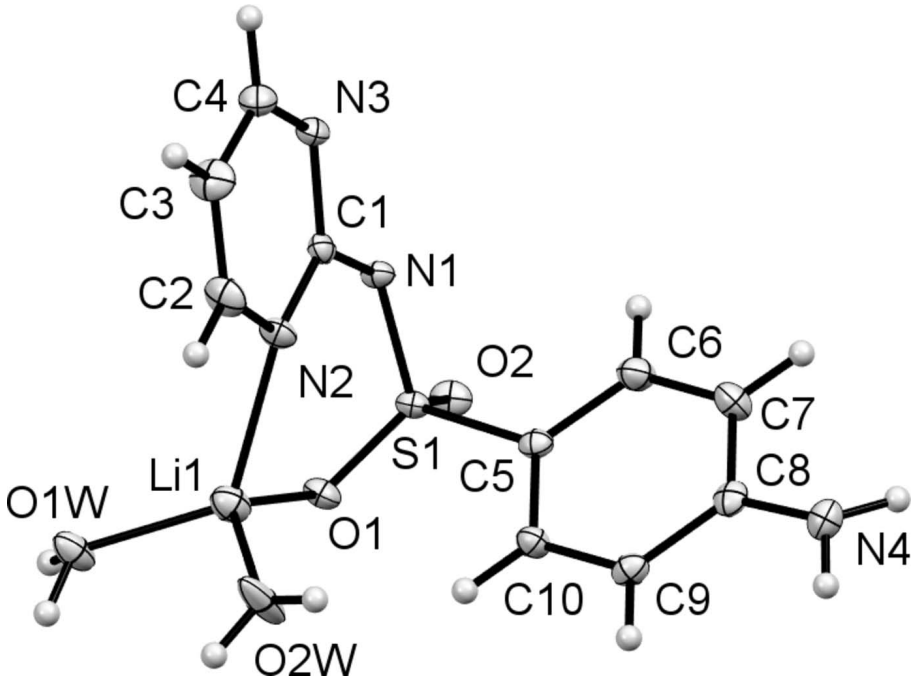


Figure 1

Molecular structure of the Li salt (I) with non-H atoms shown as 50% probability ellipsoids.

fig2.tif

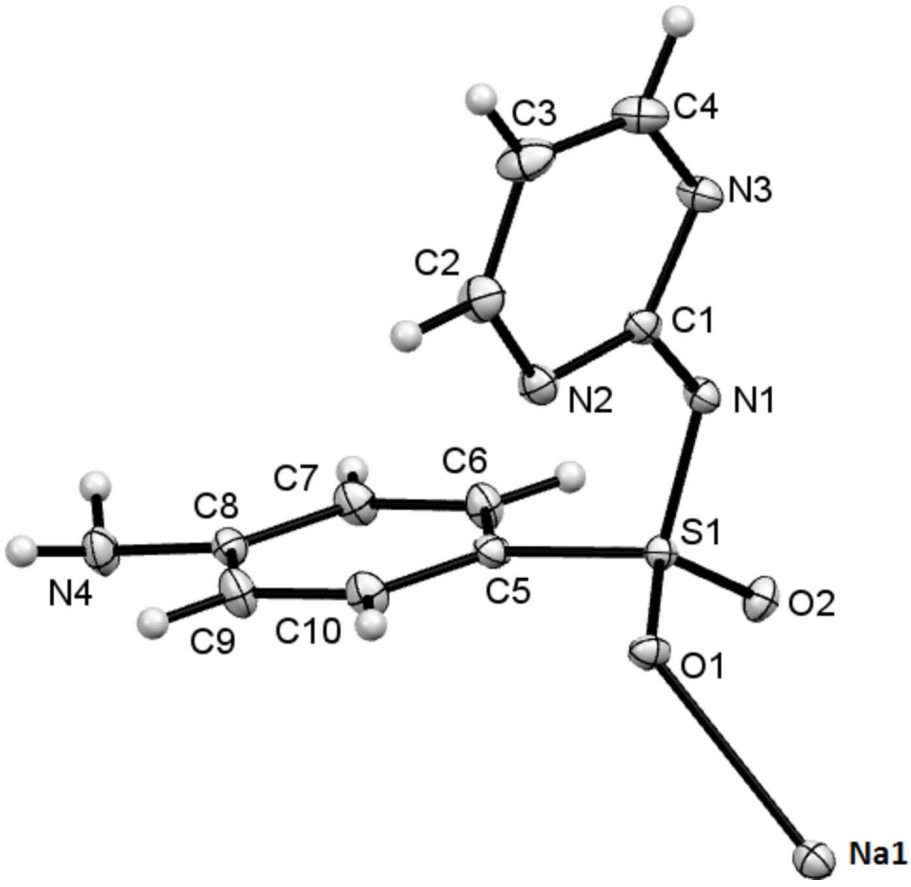


Figure 2

Contents of the asymmetric unit of the Na salt (II) with non-H atoms shown as 50% probability ellipsoids.

fig3.tif

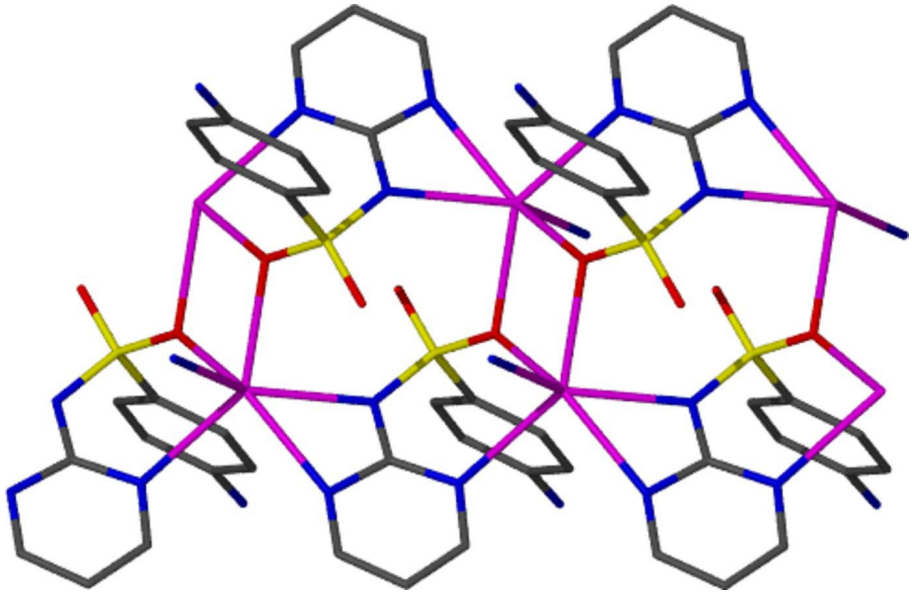


Figure 3

Part of the extended structure of (II) showing the dative bonds that give a one-dimensional chain that extends in the crystallographic *a* direction. H atoms are omitted for clarity. Here and in other colour figures, black = C, blue = N, red = O, yellow = S, pink = alkali metal.

fig4.tif

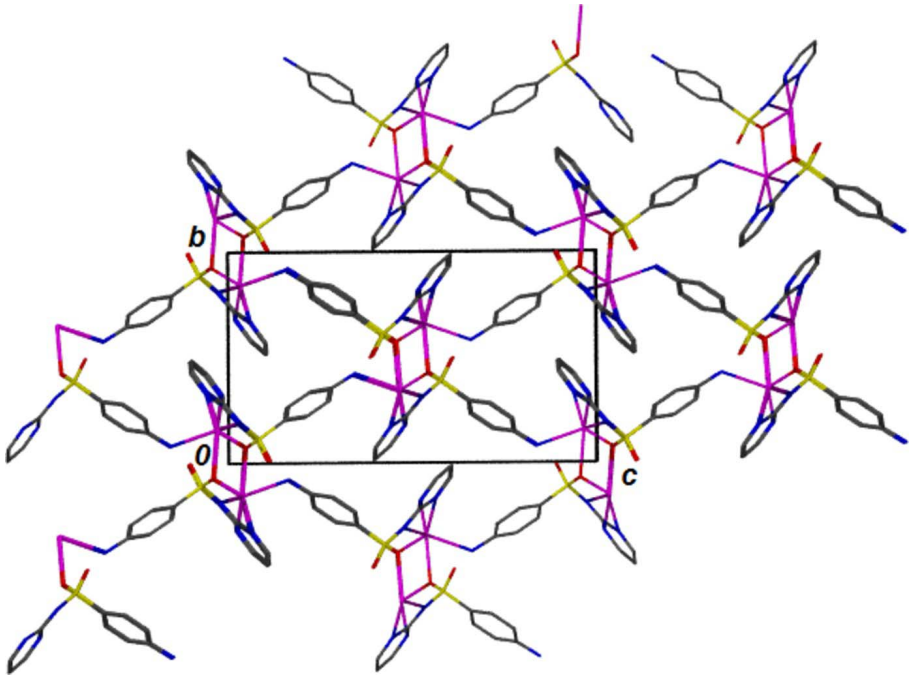


Figure 4

Packing diagram for (II) with view down the *a* axis direction.

fig5.tif

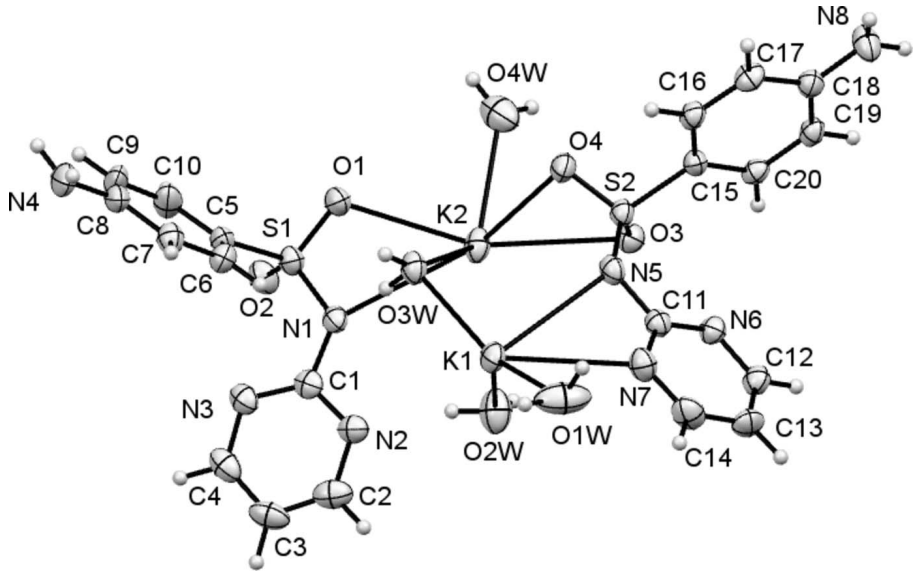


Figure 5

Contents of the asymmetric unit of the K salt (III) with non-H atoms shown as 50% probability ellipsoids. Disordered H atom positions on water ligand O4W are not shown.

fig6.tif

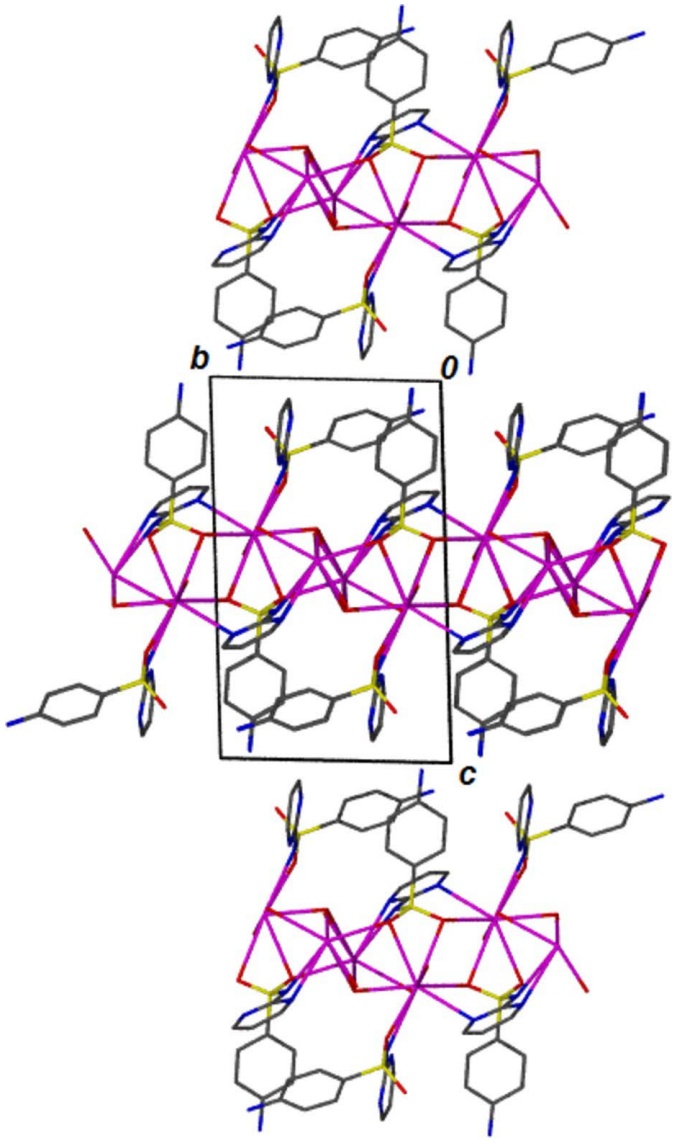
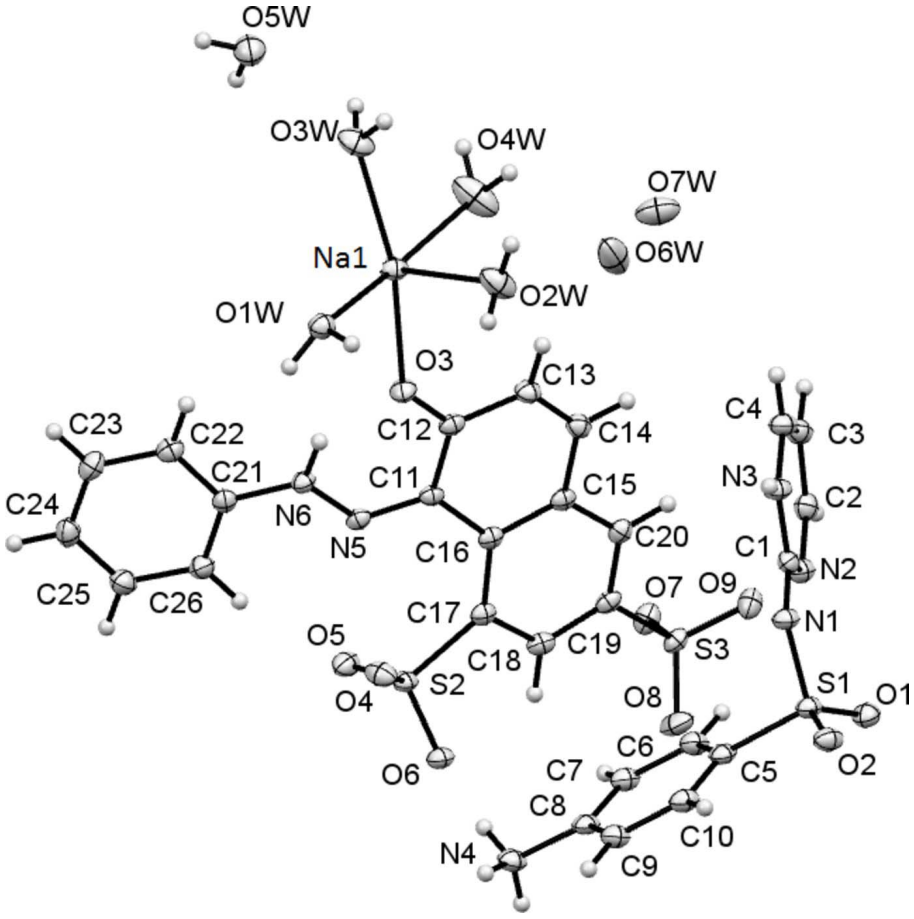


Figure 6

Packing diagram for showing the layered structure of (III) with view down the *a* axis direction.

fig7.tif



160 **Figure 7**

161 Contents of the asymmetric unit of (IV) with non-H atoms shown as 50% probability ellipsoids. Disordered H atom

162 positions on water molecules are not shown.

fig8.tif

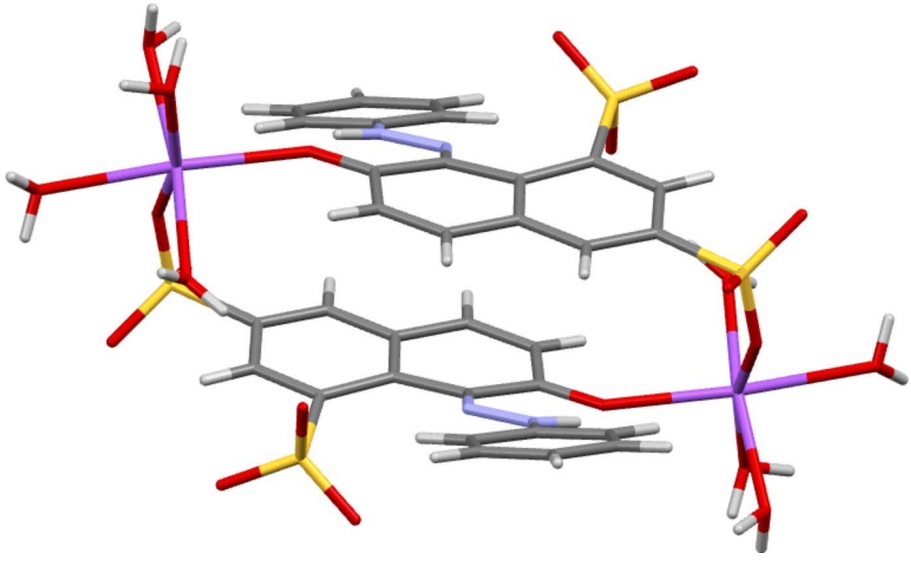


Figure 8

View of the dimeric $[Na(H_2O)_4]_2[OG]_2$ fragment found in structure (IV).

fig9.tif

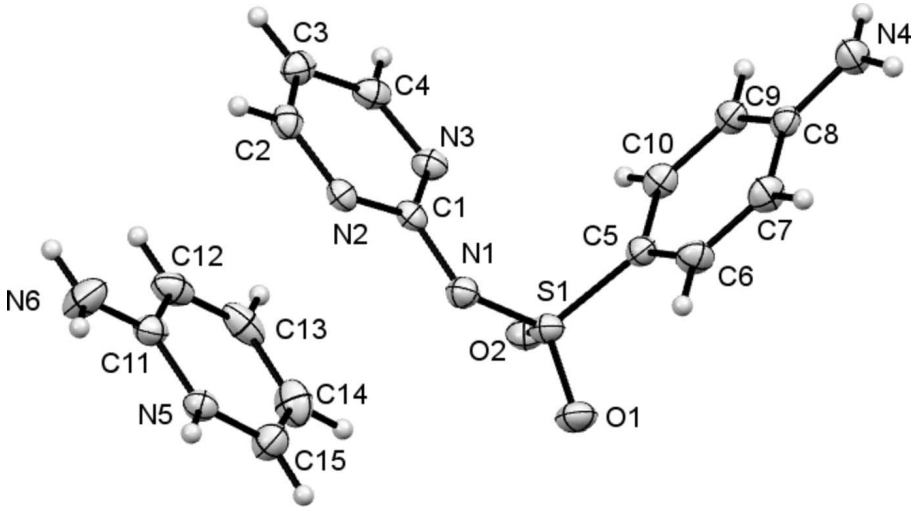


Figure 9

Contents of the asymmetric unit of (V) with non-H atoms shown as 50% probability ellipsoids.

fig10.tif

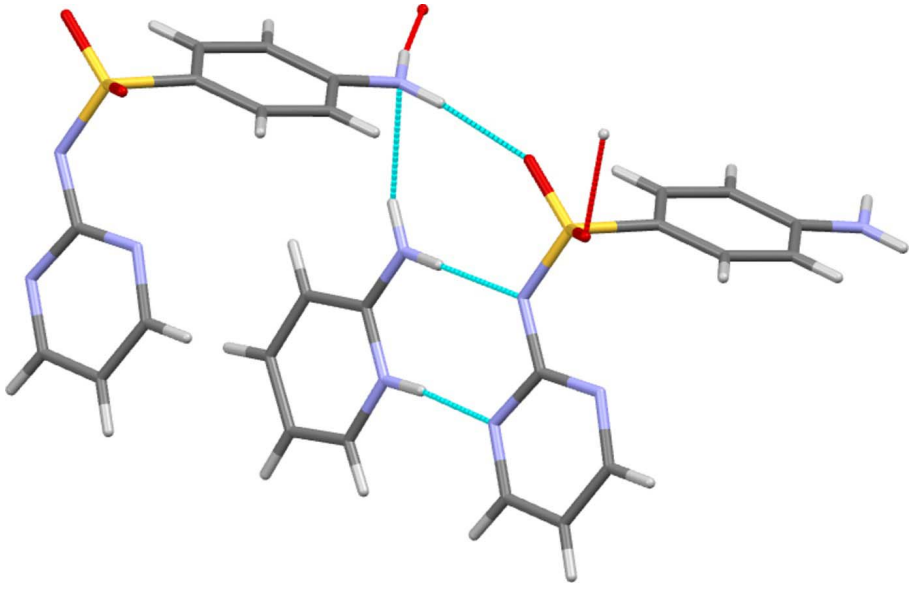
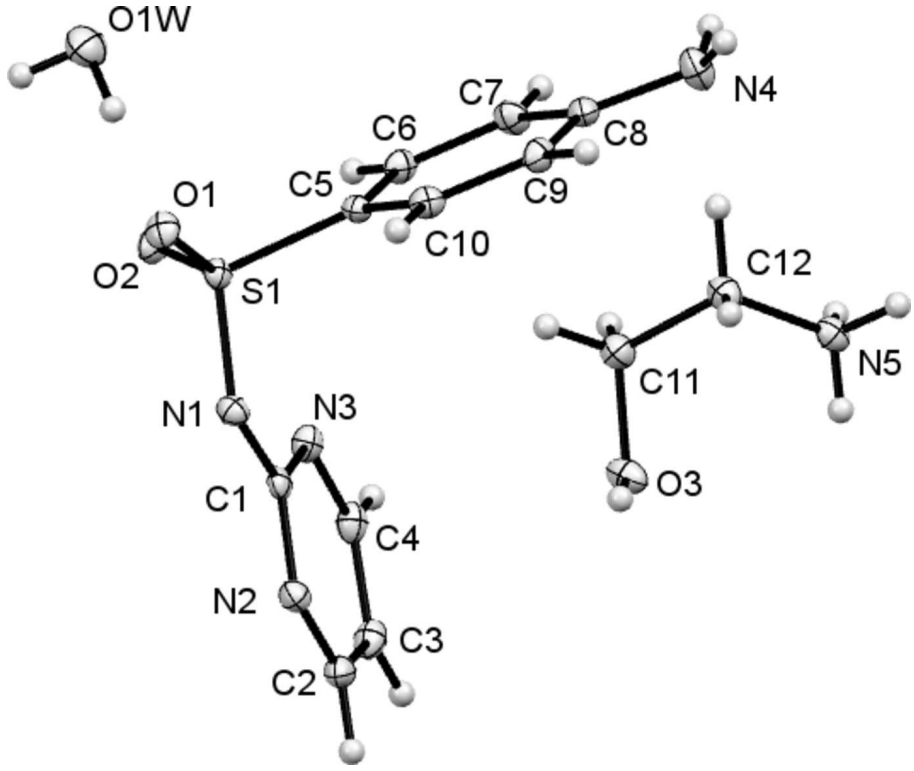


Figure 10

Detail from the structure of (V) highlighting the $R_2^2(8)$ cation to anion hydrogen bond interaction and the role of the amine groups.

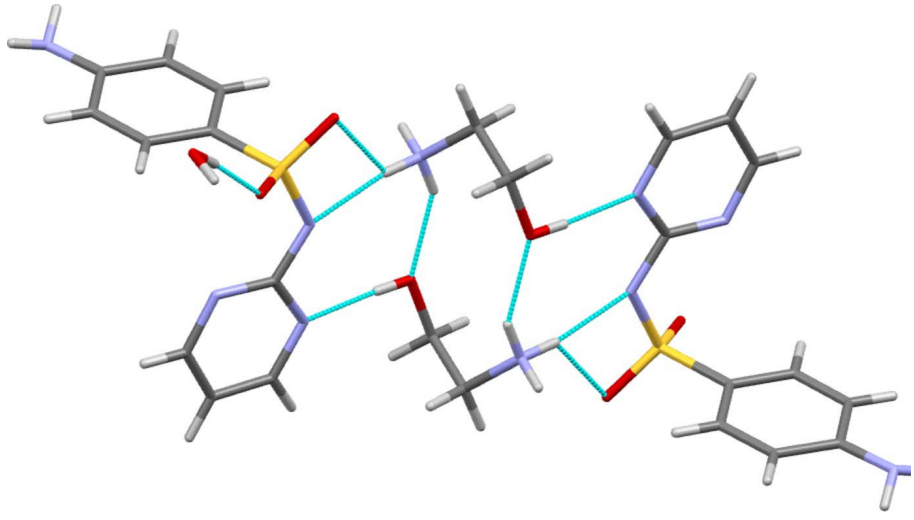
fig11.tif



170 **Figure 11**

171 Contents of the asymmetric unit of (VI) with non-H atoms shown as 50% probability ellipsoids.

fig12.tif



172 **Figure 12**

173 Detail from the structure of (VI) showing the hydrogen bonded tetramer formed by two cations and two anions.

174 **Table 1**

175 Experimental details

176	(I)	(II)	(III)	(IV)
177	Crystal data			

178	Chemical formula	$C_{10}H_{13}LiN_4O_4S$	$C_{10}H_9N_4NaO_2S$	$C_{10}H_{13}KN_4O_4S$	$C_{26}H_{32}N_6NaO_{14.50}S_3$
179	M_r	292.24	272.26	324.40	779.74
180	Crystal system, space group	Monoclinic, *Cc*	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$
181	Temperature (K)	123	123	123	100
182	*a*, *b*, *c* (Å)	11.5095 (4), 12.0788 (5), 9.5184 (4)	5.9010 (11), 10.534 (4), 18.389 (3)	8.8503 (7), 9.6385 (4), 15.9734 (7)	12.7688 (1), 16.9040 (1), 15.7411 (1)
183	α, β, γ (°)	90, 91.063 (3), 90	90, 94.216 (15), 90	92.350 (4), 95.186 (4), 94.209 (4)	90, 107.609 (1), 90
184	*V* (Å^3)	1323.03 (9)	1140.0 (5)	1351.79 (13)	3238.42 (4)
185	*Z*	4	4	4	4
186	Radiation type	Mo *Kα*	Mo *Kα*	Cu *Kα*	Cu *Kα*
187	μ (mm^{-1})	0.26	0.32	5.09	2.95
188	Crystal size (mm)	0.30 × 0.20 × 0.18	0.35 × 0.12 × 0.08	0.28 × 0.15 × 0.10	0.12 × 0.08 × 0.04
189					
190	Data collection				
191	Diffractometer	Oxford Diffraction Xcalibur E	Oxford Diffraction Xcalibur E	Oxford Diffraction Gemini S	XtaLAB AFC11 (RCD3)
192	Absorption correction	Multi-scan *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan *CrysAlis PRO* 1.171.39.34b (Rigaku Oxford Diffraction, 2017) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
193	T_{min}, T_{max}	0.949, 1.000	0.961, 1.000	0.601, 1.000	0.615, 1.000
194	No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3390, 2246, 2184	5903, 2786, 2351	15580, 5357, 3951	59125, 5913, 5686
195	R_{int}	0.016	0.029	0.050	0.027
196	(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.682	0.693	0.623	0.602
197					
198	Refinement				
199	$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.026, 0.066, 1.04	0.038, 0.088, 1.06	0.060, 0.157, 1.05	0.028, 0.075, 1.05
200	No. of reflections	2246	2786	5357	5913
201	No. of parameters	207	171	404	537
202	No. of restraints	8	0	14	13
203	H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
204	$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.25	0.43, −0.46	0.81, −0.45	0.26, −0.39

Absolute structure	Refined as an inversion twin.	?	?	?
Absolute structure parameter	−0.01 (8)	?	?	?

	(V)	(VI)
Crystal data		
Chemical formula	$C_{15}H_{16}N_6O_2S$	$C_{12}H_{19}N_5O_4S$
M_r	344.40	329.38
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	123	123
a, b, c (Å)	8.5796 (2), 19.0371 (5), 11.2512 (4)	12.7755 (4), 9.8979 (4), 11.5366 (4)
α, β, γ (°)	90, 121.116 (3), 90	90, 93.508 (3), 90
V (Å^3)	1573.27 (9)	1456.08 (9)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.23	0.25
Crystal size (mm)	0.4 × 0.3 × 0.02	0.25 × 0.24 × 0.12
Data collection		
Diffractometer	Oxford Diffraction Xcalibur E	Oxford Diffraction Xcalibur E
Absorption correction	Multi-scan *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min}, T_{max}	0.527, 1.000	0.904, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22193, 4082, 3225	7213, 3571, 2942
R_{int}	0.040	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.682	0.694
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.114, 1.04	0.037, 0.095, 1.06
No. of reflections	4082	3571
No. of parameters	237	231
No. of restraints	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.48	0.45, −0.47
Absolute structure	?	?

Absolute structure parameter? ?

Computer programs: *CrysAlis PRO* (Agilent, 2014), *CrysAlis PRO*, *SIR92* (Altomare *et al.*, 1994), *SHELXS* (Sheldrick, 2015), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008), *SHELXL2014*.

Table 2

Selected geometric parameters (Å, º) for (I)

Li1—O2W	1.870 (4)	Li1—O1	1.934 (5)
Li1—O1W	1.910 (5)	Li1—N2	2.077 (5)
O2W—Li1—O1W	101.5 (2)	O2W—Li1—N2	107.1 (2)
O2W—Li1—O1	126.9 (2)	O1W—Li1—N2	124.1 (2)
O1W—Li1—O1	108.5 (2)	O1—Li1—N2	90.91 (18)

Table 3

Hydrogen-bond geometry (Å, º) for (I)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···O2^{i}	0.875 (12)	2.051 (16)	2.881 (2)	158 (3)
O2W—H3W···N1^{ii}	0.865 (12)	1.949 (15)	2.805 (3)	170 (4)
O1W—H2W···N3^{ii}	0.867 (12)	1.970 (12)	2.837 (3)	178 (3)
O2W—H4W···N4^{iii}	0.863 (12)	2.006 (12)	2.866 (3)	174 (4)
N4—H1N···O2^{iv}	0.84 (3)	2.40 (3)	3.070 (3)	137 (3)

Symmetry codes: (i) x, $-y+1$, $z-1/2$; (ii) $x+1/2$, $-y+1/2$, $z-1/2$; (iii) x, $-y$, $z-1/2$; (iv) $x+1/2$, $y-1/2$, z.

Table 4

Selected bond lengths (Å) for (II)

Na1—O1^{i}	2.2897 (15)	Na1—N3^{ii}	2.4836 (18)
Na1—N1^{ii}	2.4533 (15)	Na1—N4^{iii}	2.5771 (19)
Na1—O1	2.4647 (17)	Na1—N2^{i}	2.6670 (17)

Symmetry codes: (i) $-x+1$, $-y$, $-z$; (ii) $-x$, $-y$, $-z$; (iii) $-x+1$, $y-1/2$, $-z+1/2$.

Table 5

Hydrogen-bond geometry (Å, º) for (II)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1N···O2^{iv}	0.86 (2)	2.06 (2)	2.907 (2)	166 (2)
N4—H2N···O1^{v}	0.91 (2)	2.52 (2)	2.959 (2)	110.5 (17)

Symmetry codes: (iv) $-x$, $y+1/2$, $-z+1/2$; (v) $-x+1$, $y+1/2$, $-z+1/2$.

Table 6

Selected bond lengths (Å) for (III)

K1—O4^{i}	2.729 (3)	K2—O3^{iii}	2.759 (3)
K1—O3W	2.771 (3)	K2—O4W	2.820 (4)

K1—O1W^{ii}	2.812 (5)	K2—O3W	2.871 (3)
K1—O2W	2.824 (4)	K2—O1	2.883 (3)
K1—N7	2.932 (4)	K2—N6^{iii}	2.899 (4)
K1—O1W	2.975 (5)	K2—O4	2.947 (3)
K1—O3W^{i}	3.004 (3)	K2—O3	3.023 (3)
K1—N5	3.129 (4)	K2—N1	3.153 (3)

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x, -y+2, -z+1$.

Table 7

Hydrogen-bond geometry (Å, º) for (III)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···O1^{i}	0.880 (10)	1.93 (2)	2.799 (5)	167 (8)
O1W—H2W···O4W^{i}	0.877 (10)	2.27 (4)	3.070 (6)	151 (6)
O2W—H3W···O3^{iii}	0.878 (10)	2.071 (19)	2.933 (5)	167 (6)
O2W—H4W···N2	0.880 (10)	1.965 (17)	2.828 (5)	167 (6)
O3W—H5W···N1	0.874 (10)	2.022 (18)	2.872 (5)	164 (5)
O3W—H6W···N5^{i}	0.873 (10)	1.979 (14)	2.835 (5)	166 (4)
O4W—H7W···O2W^{iv}	0.878 (10)	2.052 (17)	2.919 (6)	169 (7)
O4W—H8W···O4W^{v}	0.879 (10)	2.05 (2)	2.920 (8)	168 (9)
N4—H1N···N4^{vi}	0.87 (7)	2.50 (6)	3.054 (9)	122 (5)
N4—H2N···O2^{vii}	0.93 (7)	2.57 (7)	3.431 (7)	153 (5)
N8—H3N···O2^{viii}	0.87 (5)	2.38 (5)	3.046 (5)	134 (4)
N8—H3N···N3^{viii}	0.87 (5)	2.61 (5)	3.283 (6)	135 (4)

Symmetry codes: (i) $-x, -y+1, -z+1$; (iii) $-x, -y+2, -z+1$; (iv) $x-1, y, z$; (v) $-x-1, -y+2, -z+1$; (vi) $-x-1, -y, -z$; (vii) $x, y-1, z$; (viii) $x, y, z+1$.

Table 8

Selected geometric parameters (Å, º) for (IV)

Na1—O1W	2.3184 (13)	Na1—O2W	2.4764 (14)
Na1—O4W	2.3211 (15)	O3—C12	1.2626 (18)
Na1—O3	2.4042 (12)	N5—N6	1.3031 (18)
Na1—O7^{i}	2.4237 (12)	N5—C11	1.3322 (19)
Na1—O3W	2.4592 (14)	C11—C12	1.467 (2)
O1W—Na1—O4W	169.11 (5)	O3—Na1—O3W	166.97 (5)
O1W—Na1—O3	90.96 (4)	O7^{i}—Na1—O3W	80.17 (5)
O4W—Na1—O3	98.12 (5)	O1W—Na1—O2W	81.14 (5)
O1W—Na1—O7^{i}	85.13 (4)	O4W—Na1—O2W	91.10 (6)
O4W—Na1—O7^{i}	101.30 (5)	O3—Na1—O2W	101.64 (5)
O3—Na1—O7^{i}	86.81 (4)	O7^{i}—Na1—O2W	163.94 (5)
O1W—Na1—O3W	87.73 (5)	O3W—Na1—O2W	90.97 (5)
O4W—Na1—O3W	84.75 (6)		

Symmetry code: (i) $-x, -y+1, -z$.

Table 9

Hydrogen-bond geometry (Å, º) for (IV)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···O9^{ii}	0.88 (3)	1.99 (3)	2.8404 (16)	163 (2)
O1W—H2W···O1^{ii}	0.91 (3)	2.24 (3)	2.8830 (17)	127 (2)
O1W—H2W···N2^{ii}	0.91 (3)	2.07 (3)	2.8972 (18)	150 (2)
O2W—H3W···O9^{ii}	0.86 (3)	2.05 (3)	2.8852 (18)	163 (2)
O2W—H4W···O6^{iii}	0.92 (4)	1.98 (4)	2.8932 (18)	174 (3)
O3W—H5W···O5^{iii}	0.86 (3)	1.98 (3)	2.8320 (18)	178 (3)
O3W—H6W···O5W	0.83 (4)	2.18 (3)	2.736 (3)	124 (3)
O3W—H6W···O6W^{iv}	0.83 (4)	2.09 (4)	2.853 (3)	153 (3)
O3W—H6W···O7W^{iv}	0.83 (4)	2.06 (4)	2.799 (3)	148 (3)
O4W—H7W···O5^{i}	0.879 (10)	2.242 (11)	3.1093 (19)	169 (2)
O4W—H8W···O4W^{iv}	0.883 (10)	2.00 (2)	2.825 (3)	154 (4)
O4W—H8W···O6W	0.883 (10)	2.41 (5)	2.871 (3)	113 (4)
O4W—H14W···O7W^{iv}	0.886 (10)	2.16 (3)	2.902 (3)	140 (4)
O5W—H9W···O4^{v}	0.883 (10)	1.845 (13)	2.710 (3)	166 (4)
O5W—H10W···O8^{i}	0.885 (10)	1.986 (14)	2.843 (3)	163 (3)
O6W—H11W···O2^{vi}	0.886 (10)	2.22 (2)	2.893 (3)	132 (2)
O6W—H12W···O2W	0.881 (10)	1.850 (11)	2.731 (3)	178 (5)
O7W—H13W···O8^{iii}	0.875 (10)	1.91 (2)	2.699 (3)	150 (3)
N6—H1N···O3	0.92 (2)	1.75 (2)	2.5342 (16)	142 (2)
N4—H2N···O3W^{vii}	0.92 (3)	1.93 (3)	2.839 (2)	171 (2)
N4—H3N···O6	0.91 (2)	1.92 (2)	2.8245 (18)	170 (2)
N4—H4N···O1^{viii}	0.91 (2)	2.01 (2)	2.8784 (18)	159.6 (19)
N4—H4N···O1W^{vii}	0.91 (2)	2.50 (2)	2.9386 (19)	110.5 (16)
N3—H5N···O2^{vi}	0.84 (2)	2.57 (2)	3.0827 (17)	120.3 (18)
N3—H5N···N1^{vi}	0.84 (2)	2.02 (2)	2.8655 (19)	177 (2)

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x, y-1/2, -z+1/2$; (iii) $x-1, y, z$; (iv) $-x-1, -y+1, -z$; (v) $x-1, -y+1/2, z-1/2$; (vi) $-x, -y+1, -z+1$; (vii) $x+1, -y+1/2, z+1/2$; (viii) $-x+1, -y+1, -z+1$.

Table 10

Hydrogen-bond geometry (Å, º) for (V)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1N···O2^{i}	0.86 (2)	2.04 (2)	2.895 (2)	168 (2)
N4—H2N···O1^{ii}	0.87 (2)	2.03 (2)	2.887 (2)	174 (2)
N5—H3N···N2^{iii}	0.92 (2)	1.85 (2)	2.758 (2)	174 (2)
N6—H4N···N4^{iv}	0.87 (3)	2.39 (3)	3.085 (2)	137 (2)
N6—H5N···N1^{iii}	0.92 (3)	2.00 (3)	2.918 (2)	173 (3)

Symmetry codes: (i) $x-1, -y+3/2, z-1/2$; (ii) $x-1, y, z$; (iii) $-x+2, -y+2, -z$; (iv) $-x+1, -y+2, -z$.

Table 11

Hydrogen-bond geometry (Å, º) for (VI)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1N···O1W^{i}	0.89 (2)	2.34 (2)	3.173 (2)	156 (2)
N4—H2N···O1W^{ii}	0.83 (2)	2.58 (2)	3.293 (2)	144.1 (19)
N5—H3N···O3^{iii}	0.86 (2)	2.01 (2)	2.7762 (18)	147.2 (18)
N5—H4N···O2^{ii}	0.90 (2)	1.99 (2)	2.8488 (18)	159 (2)
N5—H4N···N3^{ii}	0.90 (2)	2.51 (2)	3.038 (2)	118.1 (17)
N5—H5N···O1^{iv}	0.93 (2)	2.44 (2)	3.1006 (18)	128.4 (17)
N5—H5N···N1^{iv}	0.93 (2)	2.02 (2)	2.933 (2)	167.3 (19)
O3—H1H···N2^{v}	0.84 (3)	1.90 (3)	2.7399 (19)	177 (2)
O1W—H1W···O2	0.97 (3)	1.94 (3)	2.9039 (17)	174 (3)
O1W—H2W···O1^{vi}	0.86 (3)	2.07 (3)	2.8997 (18)	163 (2)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) *x*, −*y*−1/2, *z*−1/2; (iii) −*x*+1, −*y*−1, −*z*+1; (iv) *x*, *y*−1, *z*; (v) −*x*+1, −*y*, −*z*+1; (vi) *x*, −*y*+1/2, *z*+1/2.

Table 12

Coordination modes of the SD anion, showing which atoms form bonds to alkali metal cations.

	(I) Li	(II) Na	(III) K L1	(III) K L2
Donor atom				
O1	yes	yes	yes	yes
O2	no	no	no	yes
N sulfamide	no	yes	yes	yes
N ring 1	yes	yes	no	yes
N ring 2	no	yes	no	yes
N amine	no	yes	no	no

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checkCIF/PLATON results for paper uk3153

checkCIF/PLATON results
Ellipsoid plot

checkCIF/PLATON results

No syntax errors found. CIF dictionary Interpreting this report

Datablock: I

Bond precision: C-C = 0.0031 A Wavelength=0.71073
Cell: a=11.5095(4) b=12.0788(5) c=9.5184(4)
alpha=90 beta=91.063(3) gamma=90
Temperature: 123 K

	Calculated	Reported
Volume	1323.03(9)	1323.03(9)
Space group	C c	C c
Hall group	C -2yc	C -2yc
Moiety formula	C10 H13 Li N4 O4 S	?
Sum formula	C10 H13 Li N4 O4 S	C10 H13 Li N4 O4 S
Mr	292.24	292.24
Dx,g cm-3	1.467	1.467
Z	4	4
Mu (mm-1)	0.262	0.262
F000	608.0	608.0
F000'	608.76	
h,k,lmax	15,16,12	15,15,12
Nref	3512[1760]	2246
Tmin,Tmax	0.939,0.954	0.949,1.000
Tmin'	0.924	

Correction method= # Reported T Limits: Tmin=0.949 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 1.28/0.64
Theta(max)= 28.976
R(reflections)= 0.0262(2184) wR2(reflections)= 0.0655(2246)
S = 1.036 Npar= 207

Alert level B

PLAT915_ALERT_3_B No Flack x Check Done: Low Friedel Pair Coverage 34 %

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) ..... Please Check
PLAT420_ALERT_2_C D-H Without Acceptor N4 --H2N Please Check

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 6 Note
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 2 Report
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... # 18 Check
O1 -S1 -LI1 1.555 1.555 1.555 32.38 Deg.
PLAT792_ALERT_1_G Model has Chirality at S1 (Polar SPGR) R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 8 Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 95 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 10 Info

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
8 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Datablock: II

Bond precision: C-C = 0.0030 A Wavelength=0.71073
Cell: a=5.9010(11) b=10.534(4) c=18.389(3)
alpha=90 beta=94.216(15) gamma=90
Temperature: 123 K

	Calculated	Reported
Volume	1140.0(5)	1140.0(5)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H9 N4 Na O2 S	?
Sum formula	C10 H9 N4 Na O2 S	C10 H9 N4 Na O2 S
Mr	272.26	272.26
Dx,g cm-3	1.586	1.586
Z	4	4
Mu (mm-1)	0.320	0.320
F000	560.0	560.0
F000'	560.80	
h,k,lmax	8,14,25	7,14,24
Nref	3168	2786
Tmin,Tmax	0.955,0.975	0.961,1.000
Tmin'	0.894	

Correction method= # Reported T Limits: Tmin=0.961 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.879
Theta(max)= 29.486
R(reflections)= 0.0376(2351) wR2(reflections)= 0.0883(2786)
S = 1.060 Npar= 171

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) ..... Please Check
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C10 H9 N4 Na O2 S
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 2.164 Check

---

Alert level G

PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 3 Info
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) . 1.33 Ratio
PLAT793_ALERT_4_G Model has Chirality at S1 (Centro SPGR) S Verify
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 352 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 10 Info

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully

3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
6 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
4 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

Datablock: III

Bond precision: C-C = 0.0064 A Wavelength=1.54180
Cell: a=8.8503(7) b=9.6385(4) c=15.9734(7)
alpha=92.350(4) beta=95.186(4) gamma=94.209(4)
Temperature: 123 K

	Calculated	Reported
Volume	1351.79(14)	1351.79(13)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C20 H26 K2 N8 O8 S2	?
Sum formula	C20 H26 K2 N8 O8 S2	C10 H13 K N4 O4 S
Mr	648.81	324.40
Dx,g cm-3	1.594	1.594
Z	2	4
Mu (mm-1)	5.085	5.085
F000	672.0	672.0
F000'	676.80	
h,k,lmax	11,12,19	10,11,19
Nref	5456	5357
Tmin,Tmax	0.460,0.601	0.601,1.000
Tmin'	0.209	

Correction method= # Reported T Limits: Tmin=0.601 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.982
Theta(max)= 73.745
R(reflections)= 0.0597(3951) wR2(reflections)= 0.1573(5357)
S = 1.045 Npar= 404

Alert level B

PLAT420_ALERT_2_B D-H Without Acceptor O4W --H9W Please Check

---

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) ..... Please Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of K1 Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00644 Ang.
PLAT420_ALERT_2_C D-H Without Acceptor N8 --H4N Please Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 6.059 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 19 Report
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/SigmaW > 10 Outliers .... 1 Check
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

---

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 13 Note
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 2 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.004 Degree
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 3 Report

```
PLAT300_ALERT_4_G Atom Site Occupancy of H8W        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H9W        Constrained at        0.5 Check
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) .       1.29 Ratio
PLAT774_ALERT_1_G Suspect X-Y Bond in CIF: K1       --K2        ..       4.24 Ang.
PLAT774_ALERT_1_G Suspect X-Y Bond in CIF: K1       --K1        ..       4.49 Ang.
PLAT774_ALERT_1_G Suspect X-Y Bond in CIF: K1       --K2        ..       4.62 Ang.
PLAT774_ALERT_1_G Suspect X-Y Bond in CIF: K2       --K1        ..       4.24 Ang.
PLAT793_ALERT_4_G Model has Chirality at S1          (Centro SPGR)          R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............        14 Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).         1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        75 Note
```

---

  0 **ALERT level A** = Most likely a serious problem - resolve or explain
  1 **ALERT level B** = A potentially serious problem, consider carefully
  8 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
 16 **ALERT level G** = General information/check it is not something unexpected

  7 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  5 ALERT type 2 Indicator that the structure model may be wrong or deficient
  6 ALERT type 3 Indicator that the structure quality may be low
  6 ALERT type 4 Improvement, methodology, query or suggestion
  1 ALERT type 5 Informative message, check

Datablock: IV

```
Bond precision:    C-C = 0.0020 A   Wavelength=1.54184
Cell: a=12.7688(1)  b=16.9040(1)  c=15.7411(1)
      alpha=90  beta=107.609(1)  gamma=90
Temperature: 100 K
```

	Calculated	Reported
Volume	3238.42(4)	3238.42(4)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C32 H36 N4 Na2 O22 S4 ?	
Sum formula	C52 H64 N12 Na2 O29 S6	C26 H32 N6 Na O14.50 S3
Mr	1559.49	779.74
Dx,g cm-3	1.599	1.599
Z	2	4
Mu (mm-1)	2.946	2.946
F000	1620.0	1620.0
F000'	1629.77	
h,k,lmax	15,20,18	15,20,18
Nref	5935	5913
Tmin,Tmax	0.754,0.889	0.615,1.000
Tmin'	0.702	

```
Correction method= # Reported   T Limits: Tmin=0.615             Tmax=1.000  AbsCorr = MULTI-SCAN
Data completeness= 0.996
Theta(max)= 68.245
R(reflections)= 0.0280(  5686) wR2(reflections)= 0.0745(  5913)
S = 1.048 Npar= 537
```

Alert level B

```
PLAT416_ALERT_2_B Short Intra D-H..H-D      H11W     ..H13W            1.38 Ang.
```

Alert level C

```
PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) ........     Please Check
```

```
PLAT222_ALERT_3_C Non-Solv.  Resd 1  H   Uiso(max)/Uiso(min) Range        4.2 Ratio
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         19 Report
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         12 Note
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...       0.50 Check
PLAT142_ALERT_4_G s.u. on b - Axis Small or Missing ..............    0.00010 Ang.
PLAT143_ALERT_4_G s.u. on c - Axis Small or Missing ..............    0.00010 Ang.
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records          3 Report
PLAT300_ALERT_4_G Atom Site Occupancy of H8W        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H14W       Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O6W        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O7W        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H12W       Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H13W       Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O5W        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H9W        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H10W       Constrained at        0.5 Check
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  3  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  4  )       100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... Resd  4          1.50 Check
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd.  #          3 Note
             H2 O
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd.  #          4 Note
             H2 O
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............         13 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          3 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ...          2 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         18 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   1 ALERT level B = A potentially serious problem, consider carefully
   3 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  23 ALERT level G = General information/check it is not something unexpected

   2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   4 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
  18 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

Datablock: V

```
Bond precision:    C-C = 0.0028 A   Wavelength=0.71073
Cell: a=8.5796(2)  b=19.0371(5)  c=11.2512(4)
      alpha=90  beta=121.116(3)  gamma=90
Temperature: 123 K
```

	Calculated	Reported
Volume	1573.27(9)	1573.27(9)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H9 N4 O2 S, C5 H7 N2	?
Sum formula	C15 H16 N6 O2 S	C15 H16 N6 O2 S
Mr	344.40	344.40
Dx,g cm-3	1.454	1.454
Z	4	4
Mu (mm-1)	0.228	0.228
F000	720.0	720.0
F000'	720.74	
h,k,lmax	11,25,15	11,25,15
Nref	4183	4082
Tmin,Tmax	0.921,0.995	0.527,1.000

Tmin' 0.913

Correction method= # Reported T Limits: Tmin=0.527 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.976
Theta(max)= 28.994
R(reflections)= 0.0445(3225) wR2(reflections)= 0.1139(4082)
S = 1.040 Npar= 237

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) ..... Please Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 2.533 Check

---

Alert level G

PLAT128_ALERT_4_G Alternate Setting for Input Space Group P21/c P21/n Note
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # 2 Note
C5 H7 N2
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 98 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 11 Info

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
5 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Datablock: VI

Bond precision: C-C = 0.0020 A Wavelength=0.71073
Cell: a=12.7755(4) b=9.8979(4) c=11.5366(4)
alpha=90 beta=93.508(3) gamma=90
Temperature: 123 K

	Calculated	Reported
Volume	1456.08(9)	1456.08(9)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H9 N4 O2 S, C2 H8 ?	
Sum formula	C12 H19 N5 O4 S	C12 H19 N5 O4 S
Mr	329.38	329.38
Dx,g cm-3	1.503	1.503
Z	4	4
Mu (mm-1)	0.250	0.250
F000	696.0	696.0
F000'	696.79	
h,k,lmax	17,13,16	17,12,15
Nref	4052	3571
Tmin,Tmax	0.939,0.970	0.904,1.000
Tmin'	0.939	

Correction method= # Reported T Limits: Tmin=0.904 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.881
Theta(max)= 29.531

```
R(reflections)= 0.0366(  2942) wR2(reflections)= 0.0953(  3571)
S = 1.058 Npar= 231
```

Alert level C

```
PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) ........     Please Check
```

Alert level G

```
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd.  #          2 Note
             C2 H8 N O
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        432 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          9 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   1 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   4 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   1 ALERT type 2 Indicator that the structure model may be wrong or deficient
   1 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

---

database duplication summary

Datablock: I

- **Chemical name =**
- *R* factor = 0.026
- Space group =
- Formula = C10 H13 Li N4 O4 S
- a=11.5095 b=12.0788 c=9.5184
- alpha=90 beta=91.063 gamma=90

Datablock: II

- **Chemical name =**
- *R* factor = 0.038
- Space group =
- Formula = C10 H9 N4 Na O2 S
- a=5.901 b=10.534 c=18.389
- alpha=90 beta=94.216 gamma=90

Datablock: III

- **Chemical name =**
- *R* factor = 0.060

- Space group =
- Formula = C10 H13 K N4 O4 S
- a=8.8503 b=9.6385 c=15.9734
- alpha=92.35 beta=95.186 gamma=94.209

Datablock: IV

- **Chemical name =**
- *R* factor = 0.028
- Space group =
- Formula = C26 H32 N6 Na O14.50 S3
- a=12.7688 b=16.904 c=15.7411
- alpha=90 beta=107.609 gamma=90

Datablock: V

- **Chemical name =**
- *R* factor = 0.045
- Space group =
- Formula = C15 H16 N6 O2 S
- a=8.5796 b=19.0371 c=11.2512
- alpha=90 beta=121.116 gamma=90

Datablock: VI

- **Chemical name =**
- *R* factor = 0.037
- Space group =
- Formula = C12 H19 N5 O4 S
- a=12.7755 b=9.8979 c=11.5366
- alpha=90 beta=93.508 gamma=90

No duplication found.

---

reference checking results

The following references were not checked in detail as they were not recognized as journal references

Agilent (2014). CrysalisPro. Agilent Technologies Ltd., Yarnton, Oxfordshire, England.

Stahl, P. H. & Wermuth, C. G. (2008). Eds. *Handbook of Pharmaceutical Salts: Properties, Selection and Use*. VHCA: Zurich.

The following references may be incorrectly formatted

Ghedini, E., Pizzolitto, C., Albore, G., Menegazzo, F., Signoretto, M., Operti, L. & Cerrato, G. (2017). *J. Sol-Gel Sci. Tech.* **83**, 618--626.
[*Unrecognized journal title.*]

Preskey, D. & Kayes, J. B. (1976). *J. Clinical Pharm.* **1**, 39--48.
[*Unrecognized journal title.*]

Results of online verification of references to IUCr journals

More than one match found for Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435--436. #ATL:SIRPOW.92 - a program for automatic solution of crystal structures by direct methods optimized for powder data. [CNOR:wi0150]# - please check.

All references appear to be cited unambiguously

Citation comments

1 date found in data_I _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

1 date found in data_II _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

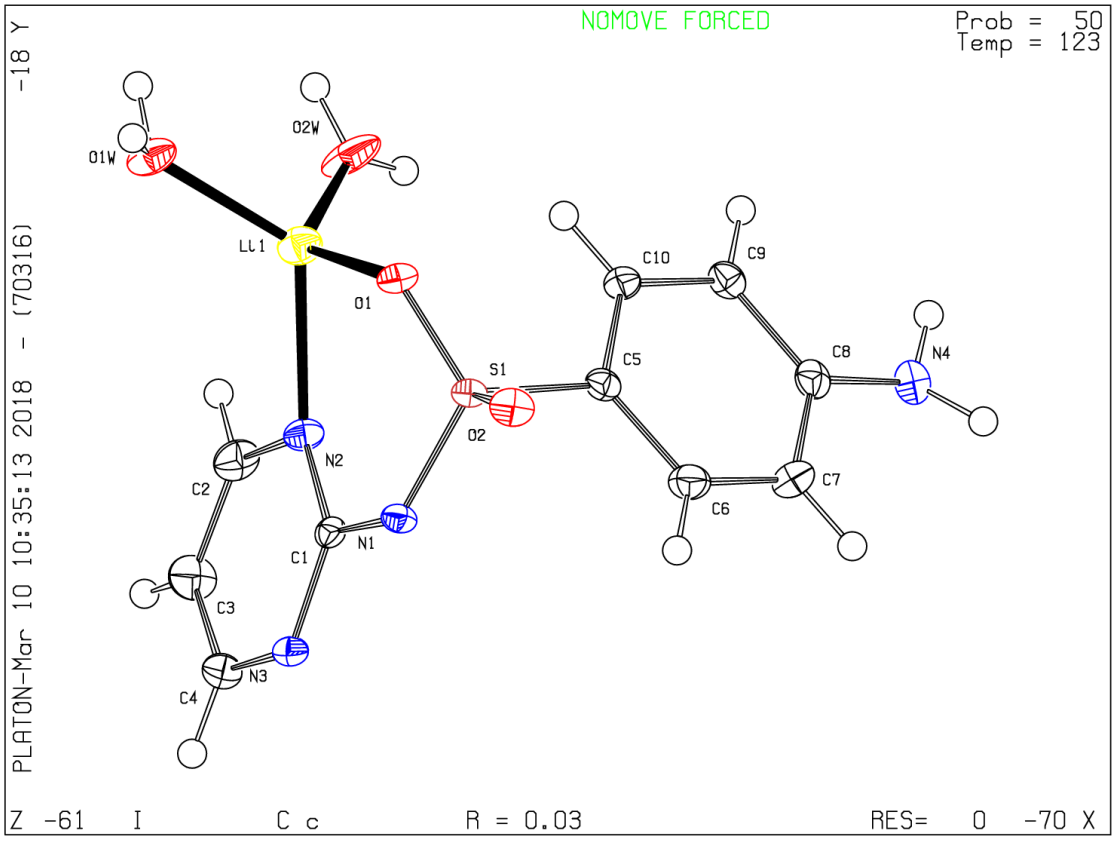
1 date found in data_III _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2014

1 date found in data_IV _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2017

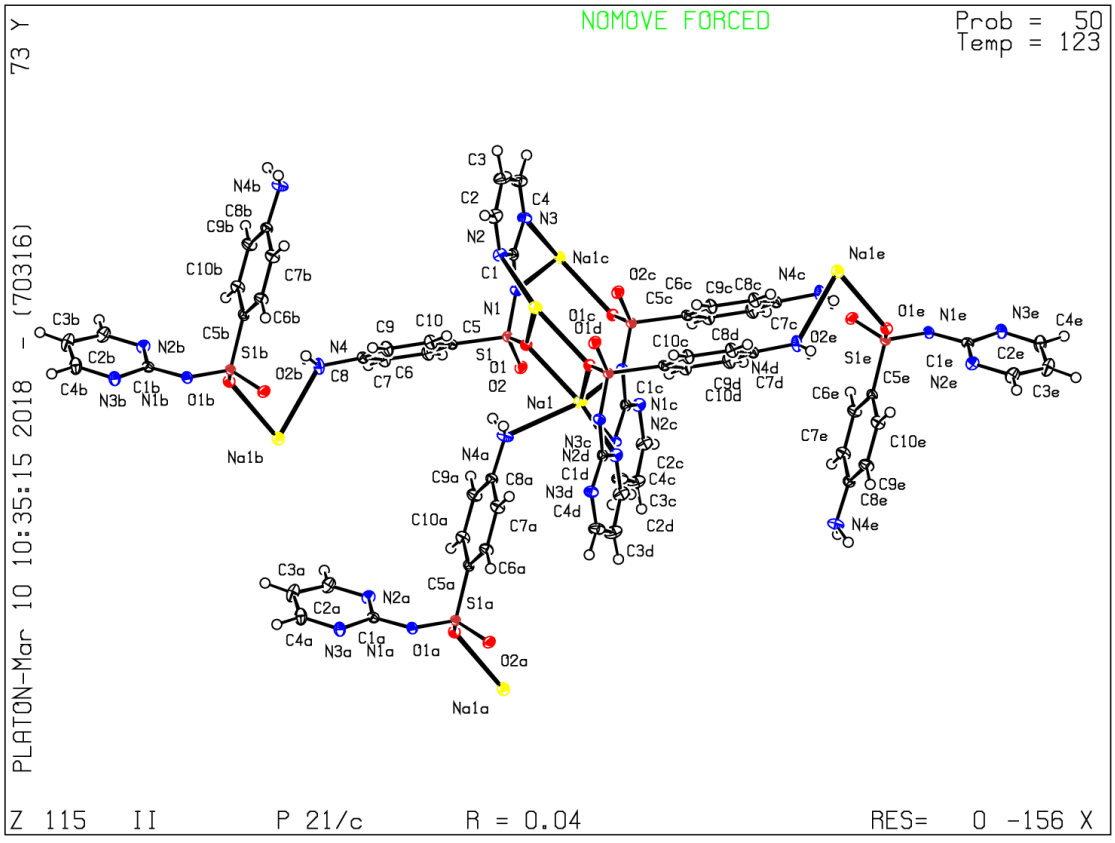
1 date found in data_V _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

1 date found in data_VI _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

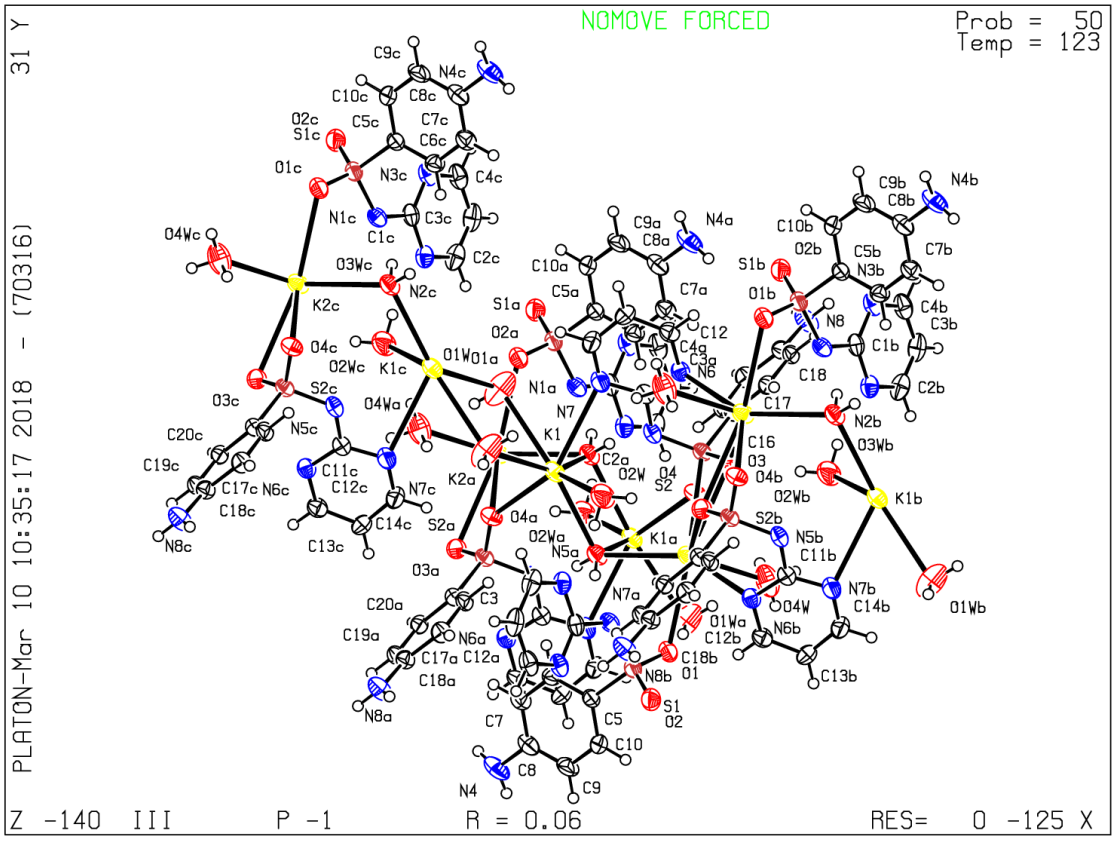
Datablock I - ellipsoid plot



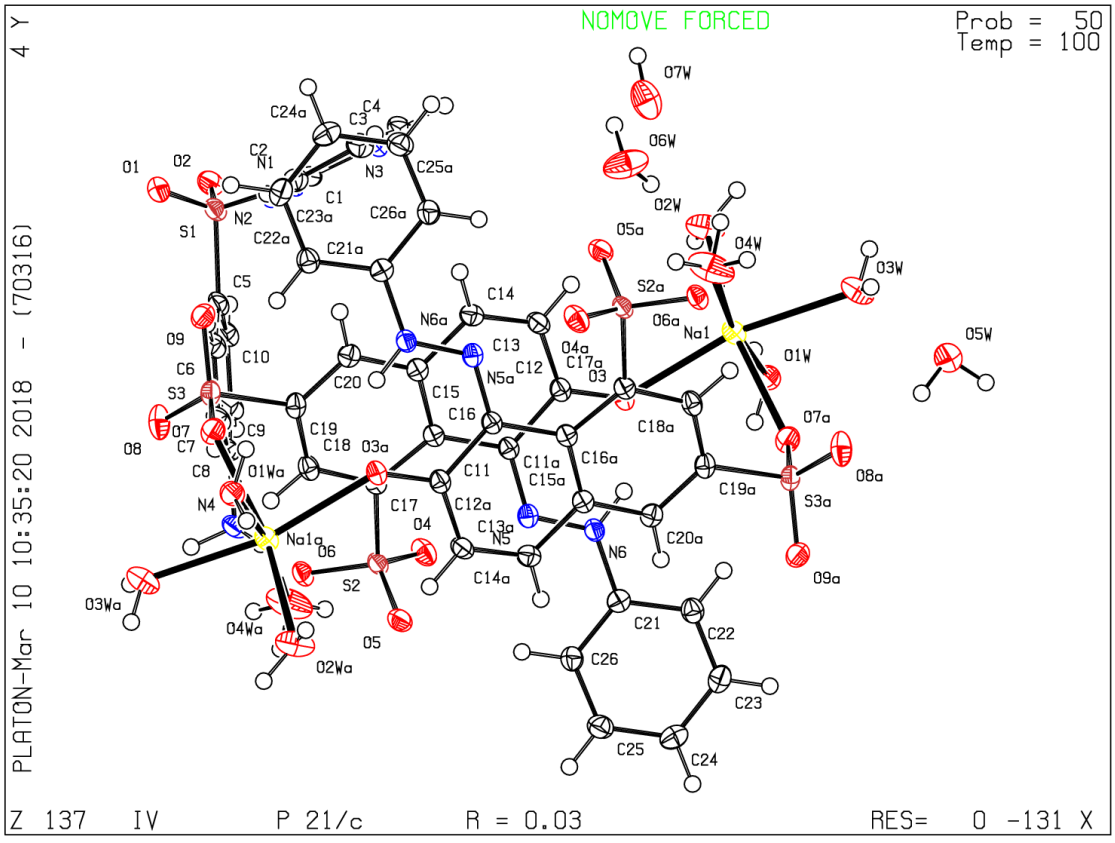
Datablock II - ellipsoid plot



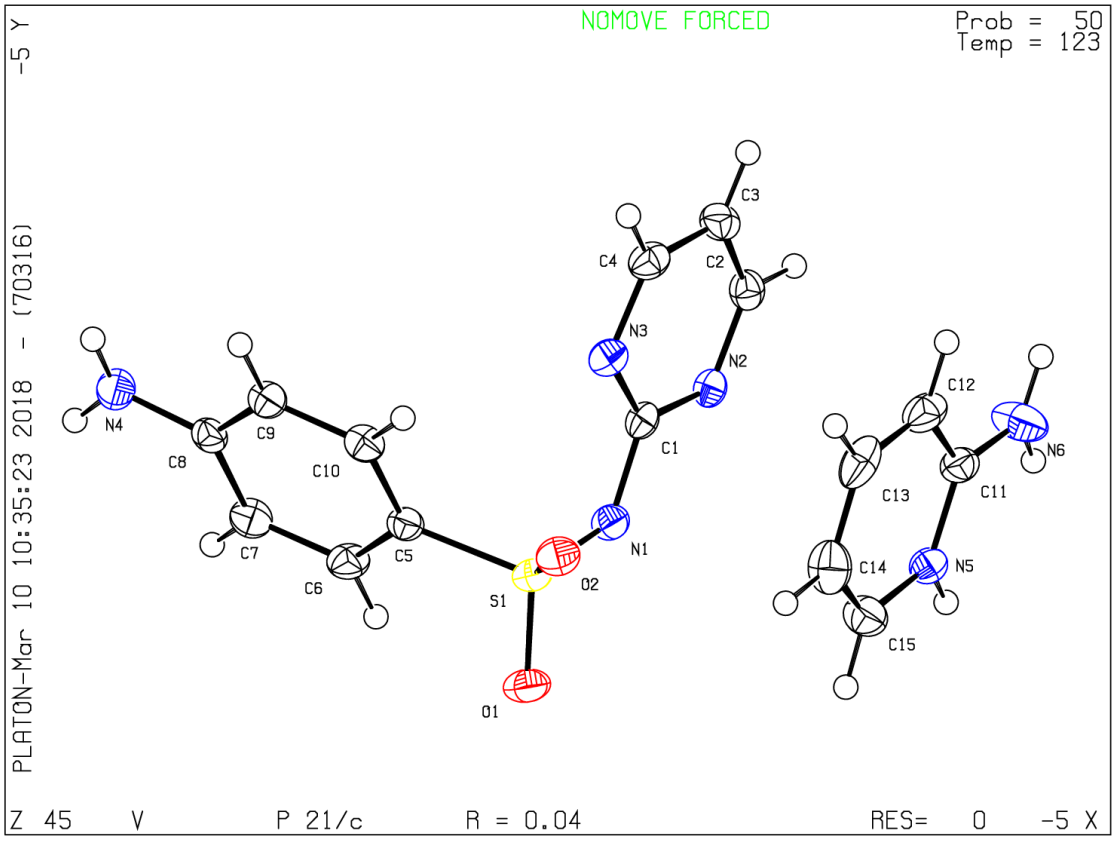
Datablock III - ellipsoid plot



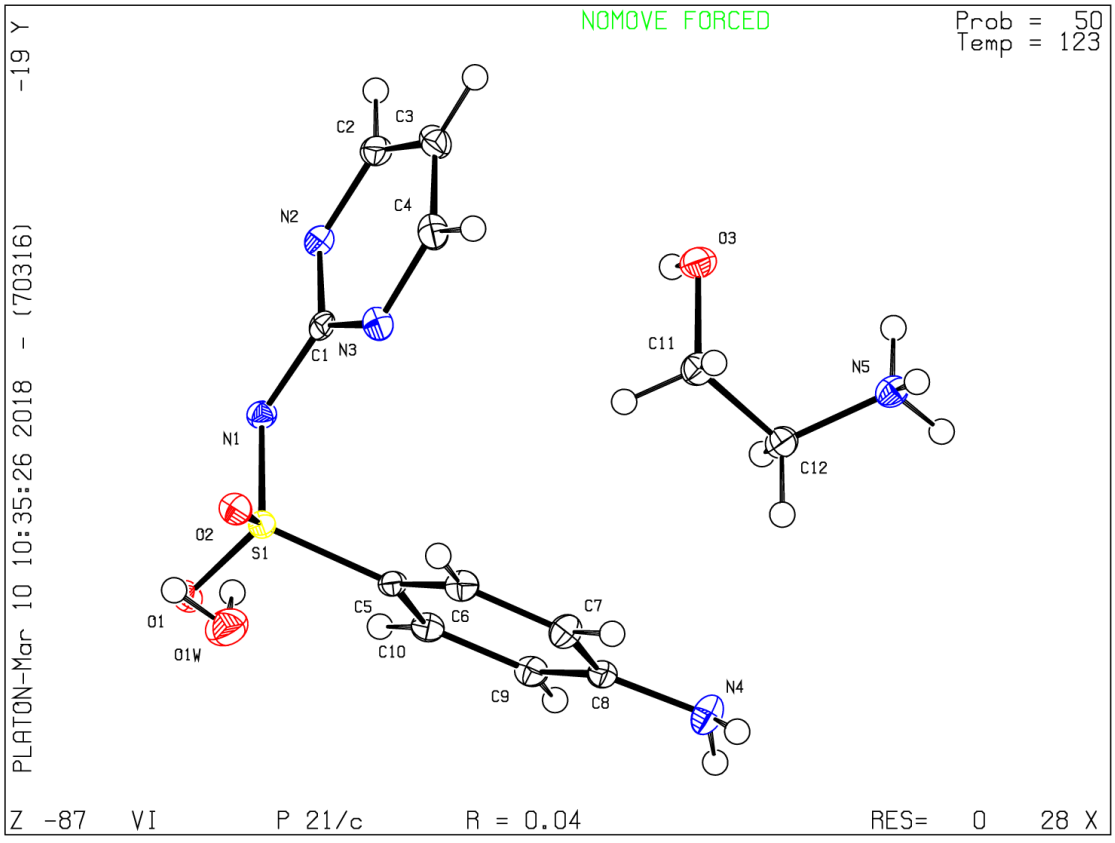
Datablock IV - ellipsoid plot



Datablock V - ellipsoid plot



Datablock VI - ellipsoid plot



supporting information

Salt forms of sulfadiazine with alkali metal and organic cations.

Gemma Campbell, Rebecca Fisher, Alan R. Kennedy,* Nathan L. C. King and Rebecca Spiteri

Computing details

For all structures, data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*. Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994) for (I), (III), (IV); *SHELXS* (Sheldrick, 2015) for (II), (V), (VI). For all structures, program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014*.

(I)

Crystal data

$C_{10}H_{13}LiN_4O_4S$
$M_r = 292.24$
Monoclinic, *Cc*
$a = 11.5095$ (4) Å
$b = 12.0788$ (5) Å
$c = 9.5184$ (4) Å
$\beta = 91.063$ (3)°
$V = 1323.03$ (9) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.467$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2629 reflections
$\theta = 3.2$–$29.8°$
$\mu = 0.26$ mm^{-1}
$T = 123$ K
Block, colourless
0.30 × 0.20 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: tube
ω scans
Absorption correction: multi-scan
CrysAlis PRO, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40)
Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
$T_{min} = 0.949$, $T_{max} = 1.000$
3390 measured reflections
2246 independent reflections
2184 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 29.0°$, $\theta_{min} = 3.2°$
$h = -15\rightarrow13$
$k = -15\rightarrow15$
$l = -12\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.066$
$S = 1.04$
2246 reflections
207 parameters
8 restraints
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2 + 0.3087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0054 (9)
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: −0.01 (8)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Li1	0.8416 (4)	0.2755 (4)	0.4134 (5)	0.0198 (8)
S1	0.75432 (4)	0.33089 (4)	0.70030 (5)	0.01208 (13)
O1	0.81653 (14)	0.36761 (14)	0.57624 (17)	0.0157 (3)
O2	0.72513 (14)	0.42112 (14)	0.79429 (18)	0.0175 (4)
O1W	0.83115 (15)	0.36603 (15)	0.24923 (19)	0.0211 (4)
O2W	0.97330 (16)	0.19039 (18)	0.3764 (2)	0.0314 (5)
N1	0.63739 (15)	0.26680 (16)	0.6691 (2)	0.0129 (4)
N2	0.71240 (17)	0.16691 (16)	0.4758 (2)	0.0154 (4)
N3	0.52506 (17)	0.13140 (17)	0.5695 (2)	0.0145 (4)
N4	1.05266 (19)	0.00920 (18)	1.0096 (2)	0.0187 (4)
C1	0.62734 (19)	0.18727 (19)	0.5688 (2)	0.0121 (4)
C2	0.6930 (2)	0.0854 (2)	0.3827 (3)	0.0206 (5)
H2	0.7523	0.0684	0.3180	0.025*
C3	0.5921 (2)	0.0254 (2)	0.3763 (3)	0.0231 (5)
H3	0.5798	−0.0317	0.3091	0.028*
C4	0.5093 (2)	0.0531 (2)	0.4735 (3)	0.0184 (5)
H4	0.4376	0.0141	0.4714	0.022*
C5	0.84694 (19)	0.23972 (19)	0.7933 (2)	0.0137 (4)
C6	0.8089 (2)	0.1949 (2)	0.9188 (3)	0.0189 (5)
H6	0.7352	0.2151	0.9541	0.023*
C7	0.8785 (2)	0.1211 (2)	0.9918 (2)	0.0198 (5)
H7	0.8523	0.0904	1.0775	0.024*
C8	0.9871 (2)	0.09088 (19)	0.9410 (2)	0.0161 (5)
C9	1.0257 (2)	0.1391 (2)	0.8169 (2)	0.0157 (5)
H9	1.1005	0.1211	0.7830	0.019*
C10	0.95556 (18)	0.21289 (19)	0.7430 (2)	0.0154 (5)
H10	0.9819	0.2450	0.6582	0.018*
H1W	0.815 (3)	0.4368 (12)	0.251 (4)	0.050 (11)*
H2W	0.890 (2)	0.365 (3)	0.194 (3)	0.055 (12)*
H3W	1.017 (3)	0.202 (3)	0.305 (3)	0.063 (12)*
H4W	1.002 (3)	0.131 (2)	0.413 (4)	0.061 (12)*

H1N	1.122 (3)	0.003 (3)	0.985 (3)	0.025 (8)*
H2N	1.044 (2)	0.012 (2)	1.107 (3)	0.017 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Li1	0.019 (2)	0.019 (2)	0.022 (2)	0.0013 (16)	0.0043 (15)	0.0013 (17)
S1	0.0108 (2)	0.0113 (2)	0.0143 (2)	−0.0013 (2)	0.00227 (16)	−0.0006 (2)
O1	0.0157 (8)	0.0145 (8)	0.0171 (8)	−0.0029 (6)	0.0045 (6)	0.0013 (7)
O2	0.0178 (8)	0.0133 (8)	0.0213 (9)	0.0001 (6)	0.0032 (6)	−0.0042 (7)
O1W	0.0208 (9)	0.0181 (9)	0.0246 (9)	0.0048 (7)	0.0098 (7)	0.0059 (8)
O2W	0.0274 (11)	0.0332 (11)	0.0345 (12)	0.0151 (8)	0.0209 (9)	0.0194 (9)
N1	0.0100 (9)	0.0144 (9)	0.0145 (9)	−0.0011 (7)	0.0020 (6)	−0.0011 (8)
N2	0.0140 (10)	0.0165 (10)	0.0160 (10)	0.0001 (8)	0.0049 (7)	−0.0007 (8)
N3	0.0135 (9)	0.0149 (10)	0.0149 (9)	−0.0035 (8)	0.0013 (7)	0.0015 (8)
N4	0.0194 (11)	0.0187 (11)	0.0180 (11)	0.0018 (8)	−0.0032 (8)	−0.0010 (8)
C1	0.0126 (10)	0.0117 (10)	0.0120 (11)	0.0003 (8)	0.0004 (7)	0.0030 (8)
C2	0.0230 (12)	0.0219 (12)	0.0172 (12)	0.0010 (10)	0.0071 (9)	−0.0048 (10)
C3	0.0262 (13)	0.0218 (12)	0.0214 (13)	−0.0044 (10)	0.0025 (9)	−0.0108 (11)
C4	0.0171 (11)	0.0182 (12)	0.0198 (12)	−0.0042 (9)	−0.0005 (8)	0.0008 (10)
C5	0.0127 (10)	0.0133 (11)	0.0150 (11)	−0.0018 (8)	0.0001 (8)	−0.0019 (9)
C6	0.0157 (11)	0.0212 (12)	0.0200 (13)	−0.0016 (10)	0.0029 (9)	0.0004 (10)
C7	0.0215 (12)	0.0208 (12)	0.0174 (12)	−0.0014 (10)	0.0049 (9)	0.0030 (10)
C8	0.0184 (11)	0.0142 (11)	0.0157 (12)	−0.0026 (9)	−0.0030 (8)	−0.0031 (9)
C9	0.0129 (11)	0.0176 (12)	0.0167 (11)	0.0018 (9)	0.0004 (8)	−0.0036 (9)
C10	0.0139 (11)	0.0177 (12)	0.0145 (11)	−0.0003 (9)	0.0027 (8)	0.0000 (9)

Geometric parameters (Å, º)

Li1—O2W	1.870 (4)	N4—C8	1.396 (3)
Li1—O1W	1.910 (5)	N4—H1N	0.84 (3)
Li1—O1	1.934 (5)	N4—H2N	0.93 (3)
Li1—N2	2.077 (5)	C2—C3	1.370 (4)
Li1—S1	3.002 (4)	C2—H2	0.9500
S1—O2	1.4533 (17)	C3—C4	1.382 (3)
S1—O1	1.4612 (16)	C3—H3	0.9500
S1—N1	1.5761 (19)	C4—H4	0.9500
S1—C5	1.760 (2)	C5—C10	1.385 (3)
O1W—H1W	0.875 (12)	C5—C6	1.390 (3)
O1W—H2W	0.867 (12)	C6—C7	1.378 (3)
O2W—H3W	0.865 (12)	C6—H6	0.9500
O2W—H4W	0.863 (12)	C7—C8	1.398 (3)
N1—C1	1.357 (3)	C7—H7	0.9500
N2—C2	1.340 (3)	C8—C9	1.397 (3)
N2—C1	1.355 (3)	C9—C10	1.386 (3)
N3—C4	1.325 (3)	C9—H9	0.9500
N3—C1	1.357 (3)	C10—H10	0.9500

O2W—Li1—O1W	101.5 (2)	C8—N4—H2N	111.7 (17)
O2W—Li1—O1	126.9 (2)	H1N—N4—H2N	113 (3)
O1W—Li1—O1	108.5 (2)	N2—C1—N3	123.4 (2)
O2W—Li1—N2	107.1 (2)	N2—C1—N1	122.3 (2)
O1W—Li1—N2	124.1 (2)	N3—C1—N1	114.3 (2)
O1—Li1—N2	90.91 (18)	N2—C2—C3	123.3 (2)
O2W—Li1—S1	125.5 (2)	N2—C2—H2	118.4
O1W—Li1—S1	126.84 (19)	C3—C2—H2	118.4
O1—Li1—S1	23.87 (8)	C2—C3—C4	115.9 (2)
N2—Li1—S1	68.13 (13)	C2—C3—H3	122.0
O2—S1—O1	113.09 (10)	C4—C3—H3	122.0
O2—S1—N1	106.15 (10)	N3—C4—C3	123.2 (2)
O1—S1—N1	115.22 (10)	N3—C4—H4	118.4
O2—S1—C5	107.73 (10)	C3—C4—H4	118.4
O1—S1—C5	107.16 (10)	C10—C5—C6	120.4 (2)
N1—S1—C5	107.14 (10)	C10—C5—S1	120.99 (18)
O2—S1—Li1	144.30 (11)	C6—C5—S1	118.58 (18)
O1—S1—Li1	32.38 (11)	C7—C6—C5	119.7 (2)
N1—S1—Li1	91.02 (11)	C7—C6—H6	120.1
C5—S1—Li1	96.20 (11)	C5—C6—H6	120.1
S1—O1—Li1	123.74 (16)	C6—C7—C8	120.7 (2)
Li1—O1W—H1W	124 (2)	C6—C7—H7	119.7
Li1—O1W—H2W	117 (2)	C8—C7—H7	119.7
H1W—O1W—H2W	101 (2)	N4—C8—C9	120.8 (2)
Li1—O2W—H3W	123 (3)	N4—C8—C7	120.1 (2)
Li1—O2W—H4W	134 (3)	C9—C8—C7	119.0 (2)
H3W—O2W—H4W	104 (2)	C10—C9—C8	120.4 (2)
C1—N1—S1	122.79 (16)	C10—C9—H9	119.8
C2—N2—C1	116.9 (2)	C8—C9—H9	119.8
C2—N2—Li1	112.7 (2)	C5—C10—C9	119.7 (2)
C1—N2—Li1	127.0 (2)	C5—C10—H10	120.1
C4—N3—C1	117.3 (2)	C9—C10—H10	120.1
C8—N4—H1N	116 (2)		

O2—S1—O1—Li1	−167.45 (18)	C2—C3—C4—N3	−1.0 (4)
N1—S1—O1—Li1	−45.1 (2)	O2—S1—C5—C10	−123.58 (19)
C5—S1—O1—Li1	74.0 (2)	O1—S1—C5—C10	−1.6 (2)
O2—S1—N1—C1	169.98 (18)	N1—S1—C5—C10	122.58 (19)
O1—S1—N1—C1	44.0 (2)	Li1—S1—C5—C10	29.6 (2)
C5—S1—N1—C1	−75.1 (2)	O2—S1—C5—C6	56.4 (2)
Li1—S1—N1—C1	21.7 (2)	O1—S1—C5—C6	178.37 (18)
C2—N2—C1—N3	−1.4 (3)	N1—S1—C5—C6	−57.4 (2)
Li1—N2—C1—N3	156.1 (2)	Li1—S1—C5—C6	−150.4 (2)
C2—N2—C1—N1	179.3 (2)	C10—C5—C6—C7	−1.7 (4)
Li1—N2—C1—N1	−23.3 (4)	S1—C5—C6—C7	178.34 (19)
C4—N3—C1—N2	0.1 (3)	C5—C6—C7—C8	0.1 (4)
C4—N3—C1—N1	179.5 (2)	C6—C7—C8—N4	−174.7 (2)
S1—N1—C1—N2	−8.8 (3)	C6—C7—C8—C9	1.8 (4)

S1—N1—C1—N3	171.78 (16)	N4—C8—C9—C10	174.3 (2)
C1—N2—C2—C3	1.5 (4)	C7—C8—C9—C10	−2.1 (3)
Li1—N2—C2—C3	−159.1 (3)	C6—C5—C10—C9	1.4 (3)
N2—C2—C3—C4	−0.4 (4)	S1—C5—C10—C9	−178.66 (17)
C1—N3—C4—C3	1.2 (4)	C8—C9—C10—C5	0.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···O2^{i}	0.88 (1)	2.05 (2)	2.881 (2)	158 (3)
O2*W*—H3*W*···N1^{ii}	0.87 (1)	1.95 (2)	2.805 (3)	170 (4)
O1*W*—H2*W*···N3^{ii}	0.87 (1)	1.97 (1)	2.837 (3)	178 (3)
O2*W*—H4*W*···N4^{iii}	0.86 (1)	2.01 (1)	2.866 (3)	174 (4)
N4—H1*N*···O2^{iv}	0.84 (3)	2.40 (3)	3.070 (3)	137 (3)

Symmetry codes: (i) *x*, −*y*+1, *z*−1/2; (ii) *x*+1/2, −*y*+1/2, *z*−1/2; (iii) *x*, −*y*, *z*−1/2; (iv) *x*+1/2, *y*−1/2, *z*.

(II)

Crystal data

$C_{10}H_9N_4NaO_2S$
$M_r = 272.26$
Monoclinic, $P2_1/c$
$a = 5.9010$ (11) Å
$b = 10.534$ (4) Å
$c = 18.389$ (3) Å
$\beta = 94.216$ (15)°
$V = 1140.0$ (5) Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.586$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2254 reflections
$\theta = 3.3$–29.4°
$\mu = 0.32$ mm^{-1}
$T = 123$ K
Rod, colourless
0.35 × 0.12 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: sealed tube
ω scans
Absorption correction: multi-scan *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
$T_{min} = 0.961$, $T_{max} = 1.000$
5903 measured reflections
2786 independent reflections
2351 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 29.5°$, $\theta_{min} = 3.5°$
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 14$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.088$
$S = 1.06$
2786 reflections
171 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 0.6871P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Na1	0.41732 (11)	−0.15699 (7)	0.03430 (4)	0.01483 (18)
S1	0.13569 (7)	0.11080 (4)	0.07836 (2)	0.01030 (12)
O1	0.34050 (19)	0.07207 (12)	0.04443 (7)	0.0130 (3)
O2	0.0133 (2)	0.00644 (12)	0.10838 (7)	0.0152 (3)
N1	−0.0383 (2)	0.18774 (14)	0.02732 (8)	0.0121 (3)
N2	0.2445 (2)	0.32085 (15)	−0.01969 (9)	0.0154 (3)
N3	−0.1495 (2)	0.33011 (16)	−0.06053 (9)	0.0155 (3)
N4	0.4138 (3)	0.41926 (17)	0.34001 (9)	0.0182 (4)
C1	0.0271 (3)	0.28102 (17)	−0.01786 (10)	0.0122 (4)
C2	0.2788 (3)	0.42162 (19)	−0.06182 (11)	0.0197 (4)
H2	0.4295	0.4527	−0.0633	0.024*
C3	0.1082 (3)	0.4826 (2)	−0.10308 (12)	0.0224 (4)
H3	0.1351	0.5566	−0.1307	0.027*
C4	−0.1042 (3)	0.4292 (2)	−0.10165 (11)	0.0198 (4)
H4	−0.2252	0.4653	−0.1318	0.024*
C5	0.2237 (3)	0.20545 (17)	0.15397 (10)	0.0121 (4)
C6	0.0767 (3)	0.21945 (18)	0.20992 (10)	0.0154 (4)
H6	−0.0670	0.1783	0.2059	0.019*
C7	0.1368 (3)	0.29163 (19)	0.27051 (10)	0.0162 (4)
H7	0.0348	0.3008	0.3078	0.019*
C8	0.3482 (3)	0.35145 (18)	0.27712 (10)	0.0141 (4)
C9	0.4953 (3)	0.33673 (19)	0.22135 (11)	0.0177 (4)
H9	0.6399	0.3768	0.2257	0.021*
C10	0.4342 (3)	0.26494 (19)	0.15998 (10)	0.0160 (4)
H10	0.5352	0.2565	0.1224	0.019*
H1N	0.301 (4)	0.454 (2)	0.3602 (13)	0.023 (6)*
H2N	0.528 (4)	0.476 (2)	0.3346 (13)	0.029 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0126 (3)	0.0166 (4)	0.0152 (4)	−0.0003 (3)	0.0003 (3)	0.0012 (3)
S1	0.0107 (2)	0.0106 (2)	0.0097 (2)	0.00025 (15)	0.00075 (15)	−0.00029 (17)
O1	0.0130 (6)	0.0136 (6)	0.0126 (6)	0.0030 (5)	0.0020 (5)	−0.0009 (5)

O2	0.0180 (6)	0.0121 (6)	0.0159 (7)	−0.0030 (5)	0.0026 (5)	0.0004 (6)
N1	0.0107 (7)	0.0137 (8)	0.0118 (8)	0.0004 (6)	−0.0005 (5)	0.0013 (6)
N2	0.0119 (7)	0.0156 (8)	0.0188 (8)	−0.0006 (6)	0.0019 (6)	0.0007 (7)
N3	0.0126 (7)	0.0173 (8)	0.0163 (8)	0.0029 (6)	−0.0002 (6)	0.0032 (7)
N4	0.0157 (8)	0.0218 (9)	0.0168 (9)	−0.0016 (7)	−0.0016 (6)	−0.0077 (7)
C1	0.0116 (8)	0.0136 (9)	0.0114 (9)	0.0017 (7)	0.0013 (6)	−0.0022 (7)
C2	0.0165 (9)	0.0183 (10)	0.0247 (11)	−0.0013 (7)	0.0052 (7)	0.0027 (9)
C3	0.0243 (10)	0.0171 (10)	0.0267 (12)	0.0027 (8)	0.0084 (8)	0.0075 (9)
C4	0.0182 (9)	0.0212 (10)	0.0203 (10)	0.0067 (8)	0.0023 (7)	0.0053 (9)
C5	0.0123 (8)	0.0131 (9)	0.0106 (9)	0.0017 (7)	−0.0014 (6)	0.0002 (7)
C6	0.0125 (8)	0.0194 (10)	0.0143 (9)	−0.0019 (7)	0.0011 (7)	−0.0023 (8)
C7	0.0132 (8)	0.0212 (10)	0.0145 (9)	−0.0003 (7)	0.0027 (7)	−0.0021 (8)
C8	0.0146 (8)	0.0143 (9)	0.0129 (9)	0.0013 (7)	−0.0024 (7)	−0.0014 (8)
C9	0.0131 (8)	0.0223 (10)	0.0174 (10)	−0.0041 (7)	−0.0010 (7)	−0.0024 (9)
C10	0.0132 (8)	0.0204 (10)	0.0148 (9)	−0.0012 (7)	0.0031 (7)	−0.0011 (8)

Geometric parameters (Å, º)

Na1—O1^{i}	2.2897 (15)	N4—C8	1.390 (2)
Na1—N1^{ii}	2.4533 (15)	N4—Na1^{iv}	2.5772 (19)
Na1—O1	2.4647 (17)	N4—H1N	0.86 (2)
Na1—N3^{ii}	2.4836 (18)	N4—H2N	0.91 (2)
Na1—N4^{iii}	2.5771 (19)	C1—Na1^{ii}	2.9255 (19)
Na1—N2^{i}	2.6670 (17)	C2—C3	1.375 (3)
Na1—C1^{ii}	2.9255 (19)	C2—H2	0.9500
Na1—Na1^{i}	3.6964 (19)	C3—C4	1.376 (3)
S1—O2	1.4470 (14)	C3—H3	0.9500
S1—O1	1.4585 (13)	C4—H4	0.9500
S1—N1	1.5652 (15)	C5—C10	1.388 (2)
S1—C5	1.7582 (18)	C5—C6	1.402 (2)
O1—Na1^{i}	2.2897 (15)	C6—C7	1.374 (3)
N1—C1	1.361 (2)	C6—H6	0.9500
N1—Na1^{ii}	2.4533 (15)	C7—C8	1.395 (2)
N2—C2	1.338 (3)	C7—H7	0.9500
N2—C1	1.353 (2)	C8—C9	1.400 (3)
N2—Na1^{i}	2.6670 (17)	C9—C10	1.384 (3)
N3—C4	1.328 (3)	C9—H9	0.9500
N3—C1	1.360 (2)	C10—H10	0.9500
N3—Na1^{ii}	2.4836 (18)		
O1^{i}—Na1—N1^{ii}	110.47 (5)	C4—N3—C1	116.73 (16)
O1^{i}—Na1—O1	78.00 (5)	C4—N3—Na1^{ii}	147.74 (13)
N1^{ii}—Na1—O1	89.87 (5)	C1—N3—Na1^{ii}	94.60 (11)
O1^{i}—Na1—N3^{ii}	147.71 (6)	C8—N4—Na1^{iv}	130.68 (13)
N1^{ii}—Na1—N3^{ii}	54.64 (5)	C8—N4—H1N	113.5 (15)
O1—Na1—N3^{ii}	125.52 (6)	Na1^{iv}—N4—H1N	90.2 (15)
O1^{i}—Na1—N4^{iii}	103.13 (6)	C8—N4—H2N	114.1 (15)
N1^{ii}—Na1—N4^{iii}	137.28 (6)	Na1^{iv}—N4—H2N	93.5 (15)

280	O1—Na1—N4^{iii}	71.83 (5)	H1N—N4—H2N	112 (2)
281	N3^{ii}—Na1—N4^{iii}	105.32 (6)	N2—C1—N3	123.76 (17)
282	O1^{i}—Na1—N2^{i}	71.51 (5)	N2—C1—N1	123.45 (15)
283	N1^{ii}—Na1—N2^{i}	122.11 (6)	N3—C1—N1	112.79 (15)
284	O1—Na1—N2^{i}	141.66 (5)	N2—C1—Na1^{ii}	168.70 (13)
285	N3^{ii}—Na1—N2^{i}	92.06 (6)	N3—C1—Na1^{ii}	57.80 (9)
286	N4^{iii}—Na1—N2^{i}	92.82 (6)	N1—C1—Na1^{ii}	56.52 (8)
287	O1^{i}—Na1—C1^{ii}	134.88 (6)	N2—C2—C3	123.65 (17)
288	N1^{ii}—Na1—C1^{ii}	27.56 (5)	N2—C2—H2	118.2
289	O1—Na1—C1^{ii}	106.06 (5)	C3—C2—H2	118.2
290	N3^{ii}—Na1—C1^{ii}	27.60 (5)	C2—C3—C4	115.45 (19)
291	N4^{iii}—Na1—C1^{ii}	121.09 (6)	C2—C3—H3	122.3
292	N2^{i}—Na1—C1^{ii}	111.85 (6)	C4—C3—H3	122.3
293	O1^{i}—Na1—Na1^{i}	40.71 (3)	N3—C4—C3	123.66 (18)
294	N1^{ii}—Na1—Na1^{i}	102.42 (5)	N3—C4—H4	118.2
295	O1—Na1—Na1^{i}	37.29 (4)	C3—C4—H4	118.2
296	N3^{ii}—Na1—Na1^{i}	155.65 (5)	C10—C5—C6	119.51 (17)
297	N4^{iii}—Na1—Na1^{i}	86.15 (5)	C10—C5—S1	121.98 (14)
298	N2^{i}—Na1—Na1^{i}	109.06 (5)	C6—C5—S1	118.51 (13)
299	C1^{ii}—Na1—Na1^{i}	128.42 (5)	C7—C6—C5	121.10 (16)
300	O2—S1—O1	113.78 (8)	C7—C6—H6	119.5
301	O2—S1—N1	107.35 (8)	C5—C6—H6	119.5
302	O1—S1—N1	114.59 (8)	C6—C7—C8	119.79 (17)
303	O2—S1—C5	104.83 (8)	C6—C7—H7	120.1
304	O1—S1—C5	106.92 (8)	C8—C7—H7	120.1
305	N1—S1—C5	108.86 (8)	N4—C8—C7	119.84 (17)
306	S1—O1—Na1^{i}	137.43 (8)	N4—C8—C9	121.08 (16)
307	S1—O1—Na1	117.97 (7)	C7—C8—C9	119.01 (17)
308	Na1^{i}—O1—Na1	102.00 (5)	C10—C9—C8	121.26 (16)
309	C1—N1—S1	122.48 (12)	C10—C9—H9	119.4
310	C1—N1—Na1^{ii}	95.92 (10)	C8—C9—H9	119.4
311	S1—N1—Na1^{ii}	138.08 (9)	C9—C10—C5	119.32 (17)
312	C2—N2—C1	116.40 (15)	C9—C10—H10	120.3
313	C2—N2—Na1^{i}	107.99 (12)	C5—C10—H10	120.3
314	C1—N2—Na1^{i}	121.33 (12)		
315				
316	O2—S1—O1—Na1^{i}	163.45 (10)	Na1^{ii}—N1—C1—N3	13.88 (16)
317	N1—S1—O1—Na1^{i}	39.38 (14)	S1—N1—C1—Na1^{ii}	162.33 (16)
318	C5—S1—O1—Na1^{i}	−81.32 (12)	C1—N2—C2—C3	−1.3 (3)
319	O2—S1—O1—Na1	5.61 (10)	Na1^{i}—N2—C2—C3	139.15 (18)
320	N1—S1—O1—Na1	−118.45 (8)	N2—C2—C3—C4	−3.2 (3)
321	C5—S1—O1—Na1	120.84 (8)	C1—N3—C4—C3	0.3 (3)
322	O2—S1—N1—C1	−170.13 (14)	Na1^{ii}—N3—C4—C3	−164.85 (18)
323	O1—S1—N1—C1	−42.71 (17)	C2—C3—C4—N3	3.8 (3)
324	C5—S1—N1—C1	76.92 (16)	O2—S1—C5—C10	141.97 (15)
325	O2—S1—N1—Na1^{ii}	−17.00 (15)	O1—S1—C5—C10	20.87 (18)
326	O1—S1—N1—Na1^{ii}	110.42 (13)	N1—S1—C5—C10	−103.42 (16)
327	C5—S1—N1—Na1^{ii}	−129.95 (13)	O2—S1—C5—C6	−37.32 (16)

C2—N2—C1—N3	5.9 (3)	O1—S1—C5—C6	−158.41 (14)
Na1^{i}—N2—C1—N3	−128.95 (16)	N1—S1—C5—C6	77.29 (16)
C2—N2—C1—N1	−173.70 (18)	C10—C5—C6—C7	0.3 (3)
Na1^{i}—N2—C1—N1	51.4 (2)	S1—C5—C6—C7	179.62 (15)
C2—N2—C1—Na1^{ii}	100.2 (7)	C5—C6—C7—C8	−0.5 (3)
Na1^{i}—N2—C1—Na1^{ii}	−34.7 (7)	Na1^{iv}—N4—C8—C7	81.0 (2)
C4—N3—C1—N2	−5.4 (3)	Na1^{iv}—N4—C8—C9	−95.8 (2)
Na1^{ii}—N3—C1—N2	166.65 (16)	C6—C7—C8—N4	−176.79 (18)
C4—N3—C1—N1	174.24 (17)	C6—C7—C8—C9	0.1 (3)
Na1^{ii}—N3—C1—N1	−13.67 (16)	N4—C8—C9—C10	177.32 (18)
C4—N3—C1—Na1^{ii}	−172.09 (19)	C7—C8—C9—C10	0.4 (3)
S1—N1—C1—N2	−4.1 (3)	C8—C9—C10—C5	−0.6 (3)
Na1^{ii}—N1—C1—N2	−166.45 (16)	C6—C5—C10—C9	0.2 (3)
S1—N1—C1—N3	176.20 (13)	S1—C5—C10—C9	−179.03 (15)

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) −*x*, −*y*, −*z*; (iii) −*x*+1, *y*−1/2, −*z*+1/2; (iv) −*x*+1, *y*+1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1*N*···O2^{v}	0.86 (2)	2.06 (2)	2.907 (2)	166 (2)
N4—H2*N*···O1^{iv}	0.91 (2)	2.52 (2)	2.959 (2)	110.5 (17)

Symmetry codes: (iv) −*x*+1, *y*+1/2, −*z*+1/2; (v) −*x*, *y*+1/2, −*z*+1/2.

(III)

Crystal data

$C_{10}H_{13}KN_4O_4S$
$M_r = 324.40$
Triclinic, $P\overline{1}$
$a = 8.8503$ (7) Å
$b = 9.6385$ (4) Å
$c = 15.9734$ (7) Å
$\alpha = 92.350$ (4)°
$\beta = 95.186$ (4)°
$\gamma = 94.209$ (4)°
$V = 1351.79$ (13) Å^3

$Z = 4$
$F(000) = 672$
$D_x = 1.594$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 3605 reflections
$\theta = 4.6$–$73.6°$
$\mu = 5.09$ mm^{-1}
$T = 123$ K
Block, colourless
0.28 × 0.15 × 0.10 mm

Data collection

Oxford Diffraction Gemini S diffractometer
Radiation source: sealed tube
ω scans
Absorption correction: multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
$T_{min} = 0.601$, $T_{max} = 1.000$
15580 measured reflections
5357 independent reflections

3951 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 73.8°$, $\theta_{min} = 4.6°$
$h = -8 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.157$
$S = 1.05$
5357 reflections
404 parameters
14 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 2.5308P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
K1	0.24189 (11)	0.55756 (9)	0.47536 (6)	0.0350 (2)	
K2	−0.15927 (12)	0.82674 (9)	0.41066 (6)	0.0365 (2)	
S1	−0.26644 (12)	0.67031 (10)	0.19910 (6)	0.0304 (2)	
S2	−0.05315 (12)	0.81132 (10)	0.61877 (6)	0.0281 (2)	
O1	−0.3523 (3)	0.6886 (3)	0.27240 (19)	0.0345 (7)	
O2	−0.3075 (4)	0.7607 (3)	0.1321 (2)	0.0398 (7)	
O1W	0.4746 (5)	0.4378 (5)	0.5915 (3)	0.0735 (14)	
H1W	0.450 (9)	0.398 (6)	0.637 (2)	0.110*	
H2W	0.481 (10)	0.362 (4)	0.560 (4)	0.110*	
O3	−0.0548 (3)	0.9494 (3)	0.58530 (18)	0.0326 (7)	
O2W	0.2375 (5)	0.8119 (4)	0.3937 (2)	0.0549 (10)	
H3W	0.196 (7)	0.891 (4)	0.398 (4)	0.082*	
H4W	0.199 (7)	0.785 (6)	0.3424 (17)	0.082*	
O4	−0.1743 (4)	0.7140 (3)	0.57871 (18)	0.0346 (7)	
O3W	−0.0485 (4)	0.5553 (3)	0.39403 (19)	0.0359 (7)	
H5W	−0.050 (6)	0.584 (4)	0.3428 (13)	0.054*	
H6W	−0.068 (6)	0.4658 (13)	0.384 (3)	0.054*	
O4W	−0.4475 (5)	0.8724 (4)	0.4641 (3)	0.0604 (11)	
H7W	−0.541 (3)	0.843 (7)	0.445 (4)	0.091*	
H8W	−0.466 (9)	0.954 (7)	0.486 (9)	0.091*	0.5
H9W	−0.436 (10)	0.819 (11)	0.507 (5)	0.091*	0.5
N1	−0.0933 (4)	0.6879 (3)	0.2369 (2)	0.0301 (8)	
N2	0.1612 (4)	0.7098 (4)	0.2256 (2)	0.0342 (8)	
N3	−0.0068 (4)	0.6541 (4)	0.1017 (2)	0.0348 (8)	
N4	−0.4201 (6)	0.0807 (5)	0.0779 (3)	0.0504 (12)	
N5	0.0982 (4)	0.7389 (4)	0.6097 (2)	0.0317 (8)	
N6	0.2546 (4)	0.9310 (3)	0.6755 (2)	0.0303 (8)	
N7	0.3546 (5)	0.7193 (4)	0.6308 (2)	0.0384 (9)	
N8	−0.1718 (6)	0.8726 (5)	0.9774 (3)	0.0441 (10)	
C1	0.0218 (5)	0.6828 (4)	0.1849 (3)	0.0309 (9)	

C2	0.2782 (6)	0.7163 (5)	0.1785 (3)	0.0433 (11)
H2	0.3781	0.7352	0.2055	0.052*
C3	0.2599 (6)	0.6967 (5)	0.0925 (3)	0.0447 (12)
H3	0.3436	0.7068	0.0593	0.054*
C4	0.1144 (6)	0.6617 (5)	0.0567 (3)	0.0406 (11)
H4	0.0987	0.6421	−0.0023	0.049*
C5	−0.3070 (5)	0.4959 (4)	0.1601 (3)	0.0299 (9)
C6	−0.2140 (5)	0.3935 (4)	0.1847 (3)	0.0312 (9)
H6	−0.1236	0.4172	0.2207	0.037*
C7	−0.2516 (5)	0.2566 (5)	0.1571 (3)	0.0353 (10)
H7	−0.1851	0.1873	0.1732	0.042*
C8	−0.3841 (5)	0.2186 (5)	0.1065 (3)	0.0377 (11)
C9	−0.4762 (6)	0.3219 (6)	0.0812 (3)	0.0480 (13)
H9	−0.5667	0.2978	0.0453	0.058*
C10	−0.4386 (5)	0.4598 (5)	0.1073 (3)	0.0436 (12)
H10	−0.5028	0.5297	0.0892	0.052*
C11	0.2388 (5)	0.8013 (4)	0.6399 (3)	0.0295 (9)
C12	0.3946 (5)	0.9760 (4)	0.7084 (3)	0.0339 (10)
H12	0.4095	1.0670	0.7344	0.041*
C13	0.5188 (5)	0.8976 (5)	0.7068 (3)	0.0384 (10)
H13	0.6166	0.9299	0.7326	0.046*
C14	0.4908 (6)	0.7693 (5)	0.6650 (3)	0.0428 (11)
H14	0.5740	0.7133	0.6603	0.051*
C15	−0.0866 (5)	0.8295 (4)	0.7260 (2)	0.0269 (8)
C16	−0.1369 (5)	0.7129 (4)	0.7677 (3)	0.0298 (9)
H16	−0.1542	0.6246	0.7382	0.036*
C17	−0.1615 (5)	0.7256 (4)	0.8511 (3)	0.0334 (10)
H17	−0.1935	0.6454	0.8794	0.040*
C18	−0.1399 (5)	0.8561 (4)	0.8955 (3)	0.0313 (9)
C19	−0.0884 (5)	0.9708 (4)	0.8523 (3)	0.0300 (9)
H19	−0.0723	1.0597	0.8810	0.036*
C20	−0.0605 (5)	0.9583 (4)	0.7689 (3)	0.0281 (9)
H20	−0.0236	1.0373	0.7410	0.034*
H1N	−0.515 (8)	0.057 (7)	0.059 (4)	0.07 (2)*
H2N	−0.383 (7)	0.010 (7)	0.111 (4)	0.06 (2)*
H3N	−0.185 (6)	0.800 (5)	1.008 (3)	0.037 (14)*
H4N	−0.156 (6)	0.955 (6)	1.004 (3)	0.053 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0413 (5)	0.0269 (5)	0.0355 (5)	−0.0036 (4)	0.0044 (4)	−0.0060 (4)
K2	0.0504 (6)	0.0234 (4)	0.0337 (5)	−0.0016 (4)	−0.0020 (4)	−0.0007 (3)
S1	0.0337 (6)	0.0274 (5)	0.0301 (5)	0.0045 (4)	0.0037 (4)	−0.0023 (4)
S2	0.0327 (5)	0.0236 (5)	0.0269 (5)	−0.0012 (4)	0.0021 (4)	−0.0022 (4)
O1	0.0344 (17)	0.0338 (16)	0.0354 (16)	0.0017 (13)	0.0071 (13)	−0.0064 (13)
O2	0.046 (2)	0.0372 (17)	0.0375 (17)	0.0119 (14)	0.0050 (14)	0.0017 (13)
O1W	0.048 (2)	0.079 (3)	0.100 (4)	0.009 (2)	0.018 (2)	0.049 (3)

O3	0.0342 (17)	0.0296 (15)	0.0339 (16)	0.0023 (13)	0.0021 (12)	0.0019 (12)
O2W	0.077 (3)	0.0357 (19)	0.050 (2)	0.0102 (19)	−0.0048 (19)	−0.0027 (16)
O4	0.0381 (17)	0.0325 (16)	0.0310 (15)	−0.0049 (13)	0.0005 (13)	−0.0043 (12)
O3W	0.0464 (19)	0.0274 (15)	0.0330 (16)	0.0014 (14)	0.0028 (14)	−0.0039 (12)
O4W	0.079 (3)	0.039 (2)	0.067 (3)	0.0007 (19)	0.031 (2)	0.0003 (18)
N1	0.036 (2)	0.0247 (17)	0.0286 (18)	−0.0033 (15)	0.0060 (15)	−0.0011 (14)
N2	0.033 (2)	0.0271 (18)	0.043 (2)	0.0030 (15)	0.0090 (16)	0.0011 (15)
N3	0.045 (2)	0.0241 (18)	0.036 (2)	0.0029 (16)	0.0113 (17)	−0.0007 (15)
N4	0.057 (3)	0.045 (3)	0.045 (3)	−0.017 (2)	0.010 (2)	−0.020 (2)
N5	0.040 (2)	0.0240 (17)	0.0315 (19)	0.0023 (15)	0.0070 (15)	−0.0046 (14)
N6	0.033 (2)	0.0240 (17)	0.0340 (19)	−0.0005 (14)	0.0040 (15)	−0.0004 (14)
N7	0.045 (2)	0.034 (2)	0.037 (2)	0.0081 (17)	0.0049 (17)	−0.0048 (16)
N8	0.071 (3)	0.031 (2)	0.031 (2)	−0.002 (2)	0.0101 (19)	−0.0008 (17)
C1	0.038 (2)	0.0164 (19)	0.039 (2)	0.0026 (17)	0.0089 (19)	0.0003 (16)
C2	0.035 (3)	0.033 (2)	0.064 (3)	0.007 (2)	0.012 (2)	0.010 (2)
C3	0.043 (3)	0.041 (3)	0.057 (3)	0.015 (2)	0.023 (2)	0.011 (2)
C4	0.059 (3)	0.029 (2)	0.038 (2)	0.009 (2)	0.017 (2)	0.0040 (18)
C5	0.028 (2)	0.032 (2)	0.029 (2)	0.0021 (17)	0.0035 (16)	−0.0066 (17)
C6	0.032 (2)	0.029 (2)	0.031 (2)	0.0000 (18)	−0.0012 (17)	−0.0006 (17)
C7	0.043 (3)	0.030 (2)	0.033 (2)	0.0007 (19)	0.0027 (19)	−0.0010 (17)
C8	0.037 (3)	0.040 (2)	0.035 (2)	−0.008 (2)	0.0099 (19)	−0.0118 (19)
C9	0.031 (3)	0.060 (3)	0.048 (3)	−0.004 (2)	−0.002 (2)	−0.028 (2)
C10	0.030 (2)	0.054 (3)	0.046 (3)	0.016 (2)	−0.004 (2)	−0.017 (2)
C11	0.035 (2)	0.027 (2)	0.028 (2)	0.0019 (17)	0.0058 (17)	0.0002 (16)
C12	0.036 (2)	0.028 (2)	0.037 (2)	−0.0023 (18)	0.0018 (19)	−0.0017 (17)
C13	0.023 (2)	0.047 (3)	0.044 (3)	0.0054 (19)	−0.0007 (18)	−0.001 (2)
C14	0.036 (3)	0.052 (3)	0.042 (3)	0.018 (2)	0.004 (2)	0.000 (2)
C15	0.028 (2)	0.026 (2)	0.027 (2)	0.0014 (16)	0.0041 (16)	−0.0019 (15)
C16	0.030 (2)	0.022 (2)	0.036 (2)	0.0004 (16)	0.0022 (17)	−0.0044 (16)
C17	0.037 (2)	0.022 (2)	0.041 (2)	−0.0012 (18)	0.0028 (19)	0.0044 (17)
C18	0.030 (2)	0.031 (2)	0.032 (2)	0.0013 (18)	0.0016 (17)	−0.0023 (17)
C19	0.029 (2)	0.023 (2)	0.036 (2)	0.0024 (17)	0.0001 (17)	−0.0061 (16)
C20	0.025 (2)	0.0224 (19)	0.036 (2)	0.0003 (16)	0.0016 (16)	0.0004 (16)

Geometric parameters (Å, º)

K1—O4^{i}	2.729 (3)	N1—C1	1.373 (5)
K1—O3W	2.771 (3)	N2—C2	1.333 (6)
K1—O1W^{ii}	2.812 (5)	N2—C1	1.343 (6)
K1—O2W	2.824 (4)	N3—C4	1.343 (6)
K1—N7	2.932 (4)	N3—C1	1.344 (6)
K1—O1W	2.975 (5)	N4—C8	1.393 (6)
K1—O3W^{i}	3.004 (3)	N4—H1N	0.87 (7)
K1—N5	3.129 (4)	N4—H2N	0.93 (7)
K1—C11	3.457 (4)	N5—C11	1.379 (5)
K1—K2^{i}	4.2385 (13)	N6—C12	1.336 (6)
K1—K1^{i}	4.491 (2)	N6—C11	1.344 (5)
K1—K2	4.6167 (15)	N6—K2^{iii}	2.899 (4)

K1—H6W	3.04 (5)	N7—C14	1.325 (6)
K2—O3^{iii}	2.759 (3)	N7—C11	1.355 (6)
K2—O4W	2.820 (4)	N8—C18	1.369 (6)
K2—O3W	2.871 (3)	N8—H3N	0.87 (5)
K2—O1	2.883 (3)	N8—H4N	0.88 (6)
K2—N6^{iii}	2.899 (4)	C2—C3	1.372 (7)
K2—O4	2.947 (3)	C2—H2	0.9500
K2—O3	3.023 (3)	C3—C4	1.373 (7)
K2—N1	3.153 (3)	C3—H3	0.9500
K2—S2	3.3840 (14)	C4—H4	0.9500
K2—C12^{iii}	3.425 (5)	C5—C6	1.378 (6)
K2—S1	3.6587 (14)	C5—C10	1.388 (6)
K2—K1^{i}	4.2385 (13)	C6—C7	1.382 (6)
K2—H5W	2.81 (5)	C6—H6	0.9500
K2—H9W	3.01 (11)	C7—C8	1.379 (6)
S1—O2	1.445 (3)	C7—H7	0.9500
S1—O1	1.464 (3)	C8—C9	1.383 (7)
S1—N1	1.589 (4)	C9—C10	1.385 (7)
S1—C5	1.769 (4)	C9—H9	0.9500
S2—O3	1.455 (3)	C10—H10	0.9500
S2—O4	1.455 (3)	C12—C13	1.380 (6)
S2—N5	1.570 (4)	C12—K2^{iii}	3.425 (5)
S2—C15	1.768 (4)	C12—H12	0.9500
O1W—K1^{ii}	2.812 (5)	C13—C14	1.376 (7)
O1W—H1W	0.880 (10)	C13—H13	0.9500
O1W—H2W	0.877 (10)	C14—H14	0.9500
O3—K2^{iii}	2.759 (3)	C15—C20	1.387 (5)
O2W—H3W	0.878 (10)	C15—C16	1.395 (6)
O2W—H4W	0.880 (10)	C16—C17	1.372 (6)
O4—K1^{i}	2.729 (3)	C16—H16	0.9500
O3W—K1^{i}	3.004 (3)	C17—C18	1.410 (6)
O3W—H5W	0.874 (10)	C17—H17	0.9500
O3W—H6W	0.873 (10)	C18—C19	1.397 (6)
O4W—H7W	0.878 (10)	C19—C20	1.378 (6)
O4W—H8W	0.879 (10)	C19—H19	0.9500
O4W—H9W	0.878 (10)	C20—H20	0.9500

O4^{i}—K1—O3W	76.14 (9)	C12^{iii}—K2—H5W	121.5 (6)
O4^{i}—K1—O1W^{ii}	91.40 (13)	S1—K2—H5W	54.1 (7)
O3W—K1—O1W^{ii}	129.93 (13)	K1^{i}—K2—H5W	60.3 (5)
O4^{i}—K1—O2W	133.37 (10)	O3^{iii}—K2—H9W	124 (2)
O3W—K1—O2W	75.00 (11)	O4W—K2—H9W	17.0 (8)
O1W^{ii}—K1—O2W	80.19 (13)	O3W—K2—H9W	111.2 (19)
O4^{i}—K1—N7	138.88 (11)	O1—K2—H9W	87.0 (19)
O3W—K1—N7	125.77 (11)	N6^{iii}—K2—H9W	91.0 (12)
O1W^{ii}—K1—N7	95.36 (15)	O4—K2—H9W	55.4 (6)
O2W—K1—N7	87.72 (11)	O3—K2—H9W	73.4 (18)
O4^{i}—K1—O1W	83.27 (12)	N1—K2—H9W	133 (2)

551	O3W—K1—O1W	152.02 (11)	S2—K2—H9W	70.2 (11)
552	O1W^{ii}—K1—O1W	68.49 (15)	C12^{iii}—K2—H9W	78.2 (7)
553	O2W—K1—O1W	132.73 (12)	S1—K2—H9W	109.0 (18)
554	N7—K1—O1W	62.11 (14)	K1^{i}—K2—H9W	67.2 (16)
555	O4^{i}—K1—O3W^{i}	76.90 (9)	H5W—K2—H9W	122 (2)
556	O3W—K1—O3W^{i}	78.00 (10)	O2—S1—O1	113.35 (19)
557	O1W^{ii}—K1—O3W^{i}	146.61 (12)	O2—S1—N1	115.66 (19)
558	O2W—K1—O3W^{i}	130.21 (12)	O1—S1—N1	104.21 (18)
559	N7—K1—O3W^{i}	75.36 (10)	O2—S1—C5	108.1 (2)
560	O1W—K1—O3W^{i}	79.04 (11)	O1—S1—C5	107.12 (18)
561	O4^{i}—K1—N5	130.33 (10)	N1—S1—C5	107.9 (2)
562	O3W—K1—N5	81.94 (10)	O2—S1—K2	118.44 (13)
563	O1W^{ii}—K1—N5	135.34 (13)	O1—S1—K2	47.64 (12)
564	O2W—K1—N5	80.19 (11)	N1—S1—K2	59.11 (13)
565	N7—K1—N5	44.19 (10)	C5—S1—K2	132.72 (15)
566	O1W—K1—N5	97.70 (13)	O3—S2—O4	112.95 (18)
567	O3W^{i}—K1—N5	55.02 (9)	O3—S2—N5	114.45 (19)
568	O4^{i}—K1—C11	145.79 (10)	O4—S2—N5	105.51 (19)
569	O3W—K1—C11	103.38 (10)	O3—S2—C15	106.97 (18)
570	O1W^{ii}—K1—C11	112.40 (14)	O4—S2—C15	106.49 (18)
571	O2W—K1—C11	76.90 (10)	N5—S2—C15	110.2 (2)
572	N7—K1—C11	22.63 (11)	O3—S2—K2	63.26 (12)
573	O1W—K1—C11	83.25 (13)	O4—S2—K2	60.28 (12)
574	O3W^{i}—K1—C11	69.73 (9)	N5—S2—K2	97.00 (14)
575	N5—K1—C11	23.50 (10)	C15—S2—K2	152.44 (15)
576	O4^{i}—K1—K2^{i}	43.70 (6)	S1—O1—K2	110.33 (15)
577	O3W—K1—K2^{i}	94.71 (7)	K1^{ii}—O1W—K1	111.51 (15)
578	O1W^{ii}—K1—K2^{i}	109.14 (9)	K1^{ii}—O1W—H1W	126 (5)
579	O2W—K1—K2^{i}	169.33 (10)	K1—O1W—H1W	122 (6)
580	N7—K1—K2^{i}	96.31 (8)	K1^{ii}—O1W—H2W	69 (6)
581	O1W—K1—K2^{i}	57.39 (8)	K1—O1W—H2W	96 (6)
582	O3W^{i}—K1—K2^{i}	42.59 (6)	H1W—O1W—H2W	98 (2)
583	N5—K1—K2^{i}	95.81 (7)	S2—O3—K2^{iii}	133.47 (17)
584	C11—K1—K2^{i}	103.32 (7)	S2—O3—K2	91.28 (13)
585	O4^{i}—K1—K1^{i}	72.58 (7)	K2^{iii}—O3—K2	114.35 (10)
586	O3W—K1—K1^{i}	40.87 (7)	K1—O2W—H3W	139 (4)
587	O1W^{ii}—K1—K1^{i}	162.68 (12)	K1—O2W—H4W	102 (4)
588	O2W—K1—K1^{i}	105.79 (10)	H3W—O2W—H4W	99 (2)
589	N7—K1—K1^{i}	101.05 (9)	S2—O4—K1^{i}	143.3 (2)
590	O1W—K1—K1^{i}	114.70 (9)	S2—O4—K2	94.33 (14)
591	O3W^{i}—K1—K1^{i}	37.13 (6)	K1^{i}—O4—K2	96.54 (9)
592	N5—K1—K1^{i}	61.97 (7)	K1—O3W—K2	109.81 (10)
593	C11—K1—K1^{i}	84.90 (8)	K1—O3W—K1^{i}	102.00 (10)
594	K2^{i}—K1—K1^{i}	63.78 (3)	K2—O3W—K1^{i}	92.33 (9)
595	O4^{i}—K1—K2	111.87 (7)	K1—O3W—H5W	114 (4)
596	O3W—K1—K2	35.80 (6)	K2—O3W—H5W	77 (3)
597	O1W^{ii}—K1—K2	128.87 (9)	K1^{i}—O3W—H5W	144 (4)
598	O2W—K1—K2	50.39 (10)	K1—O3W—H6W	99 (4)

599	N7—K1—K2	94.90 (8)	K2—O3W—H6W	149 (4)
600	O1W—K1—K2	154.32 (11)	K1^{i}—O3W—H6W	71 (3)
601	O3W^{i}—K1—K2	84.29 (7)	H5W—O3W—H6W	101 (2)
602	N5—K1—K2	56.63 (7)	K2—O4W—H7W	134 (5)
603	C11—K1—K2	72.66 (8)	K2—O4W—H8W	121 (7)
604	K2^{i}—K1—K2	119.23 (3)	H7W—O4W—H8W	99 (2)
605	K1^{i}—K1—K2	55.45 (3)	K2—O4W—H9W	94 (8)
606	O4^{i}—K1—H6W	59.8 (5)	H7W—O4W—H9W	99 (2)
607	O3W—K1—H6W	16.5 (5)	C1—N1—S1	120.6 (3)
608	O1W^{ii}—K1—H6W	127.1 (9)	C1—N1—K2	139.9 (3)
609	O2W—K1—H6W	89.3 (5)	S1—N1—K2	95.26 (15)
610	N7—K1—H6W	136.2 (8)	C2—N2—C1	116.9 (4)
611	O1W—K1—H6W	137.8 (5)	C4—N3—C1	116.1 (4)
612	O3W^{i}—K1—H6W	73.6 (8)	C8—N4—H1N	117 (4)
613	N5—K1—H6W	92.3 (8)	C8—N4—H2N	118 (4)
614	C11—K1—H6W	115.3 (8)	H1N—N4—H2N	110 (6)
615	K2^{i}—K1—H6W	80.9 (6)	C11—N5—S2	122.3 (3)
616	K1^{i}—K1—H6W	38.7 (9)	C11—N5—K1	91.7 (2)
617	K2—K1—H6W	52.1 (5)	S2—N5—K1	138.42 (19)
618	O3^{iii}—K2—O4W	118.31 (10)	C12—N6—C11	116.1 (4)
619	O3^{iii}—K2—O3W	116.43 (10)	C12—N6—K2^{iii}	101.5 (3)
620	O4W—K2—O3W	123.26 (11)	C11—N6—K2^{iii}	123.5 (3)
621	O3^{iii}—K2—O1	130.90 (9)	C14—N7—C11	116.3 (4)
622	O4W—K2—O1	80.08 (11)	C14—N7—K1	134.6 (3)
623	O3W—K2—O1	75.53 (9)	C11—N7—K1	101.0 (3)
624	O3^{iii}—K2—N6^{iii}	63.92 (9)	C18—N8—H3N	121 (3)
625	O4W—K2—N6^{iii}	74.67 (11)	C18—N8—H4N	120 (4)
626	O3W—K2—N6^{iii}	146.26 (10)	H3N—N8—H4N	117 (5)
627	O1—K2—N6^{iii}	80.79 (10)	N2—C1—N3	124.9 (4)
628	O3^{iii}—K2—O4	111.22 (9)	N2—C1—N1	113.4 (4)
629	O4W—K2—O4	72.00 (12)	N3—C1—N1	121.7 (4)
630	O3W—K2—O4	75.72 (9)	N2—C2—C3	122.6 (5)
631	O1—K2—O4	117.84 (9)	N2—C2—H2	118.7
632	N6^{iii}—K2—O4	137.47 (10)	C3—C2—H2	118.7
633	O3^{iii}—K2—O3	65.65 (10)	C2—C3—C4	116.6 (4)
634	O4W—K2—O3	81.82 (11)	C2—C3—H3	121.7
635	O3W—K2—O3	108.46 (9)	C4—C3—H3	121.7
636	O1—K2—O3	160.24 (9)	N3—C4—C3	122.8 (5)
637	N6^{iii}—K2—O3	101.97 (9)	N3—C4—H4	118.6
638	O4—K2—O3	47.93 (8)	C3—C4—H4	118.6
639	O3^{iii}—K2—N1	97.57 (9)	C6—C5—C10	119.2 (4)
640	O4W—K2—N1	126.61 (12)	C6—C5—S1	121.0 (3)
641	O3W—K2—N1	56.72 (9)	C10—C5—S1	119.7 (4)
642	O1—K2—N1	46.81 (9)	C5—C6—C7	120.2 (4)
643	N6^{iii}—K2—N1	89.55 (10)	C5—C6—H6	119.9
644	O4—K2—N1	131.79 (9)	C7—C6—H6	119.9
645	O3—K2—N1	151.48 (10)	C8—C7—C6	121.2 (5)
646	O3^{iii}—K2—S2	85.85 (7)	C8—C7—H7	119.4

O4W—K2—S2	84.25 (10)	C6—C7—H7	119.4
O3W—K2—S2	85.87 (7)	C7—C8—C9	118.4 (4)
O1—K2—S2	143.14 (7)	C7—C8—N4	120.9 (5)
N6^{iii}—K2—S2	126.55 (7)	C9—C8—N4	120.7 (5)
O4—K2—S2	25.39 (6)	C8—C9—C10	121.0 (4)
O3—K2—S2	25.46 (6)	C8—C9—H9	119.5
N1—K2—S2	139.86 (8)	C10—C9—H9	119.5
O3^{iii}—K2—C12^{iii}	86.35 (10)	C9—C10—C5	119.9 (5)
O4W—K2—C12^{iii}	61.29 (12)	C9—C10—H10	120.1
O3W—K2—C12^{iii}	136.33 (10)	C5—C10—H10	120.1
O1—K2—C12^{iii}	62.11 (10)	N6—C11—N7	124.8 (4)
N6^{iii}—K2—C12^{iii}	22.46 (10)	N6—C11—N5	121.6 (4)
O4—K2—C12^{iii}	132.77 (10)	N7—C11—N5	113.6 (4)
O3—K2—C12^{iii}	114.98 (9)	N6—C11—K1	154.2 (3)
N1—K2—C12^{iii}	85.10 (10)	N7—C11—K1	56.4 (2)
S2—K2—C12^{iii}	135.02 (8)	N5—C11—K1	64.8 (2)
O3^{iii}—K2—S1	113.47 (7)	N6—C12—C13	123.7 (4)
O4W—K2—S1	101.02 (10)	N6—C12—K2^{iii}	56.0 (2)
O3W—K2—S1	69.16 (7)	C13—C12—K2^{iii}	143.9 (3)
O1—K2—S1	22.03 (6)	N6—C12—H12	118.2
N6^{iii}—K2—S1	79.88 (7)	C13—C12—H12	118.2
O4—K2—S1	131.84 (6)	K2^{iii}—C12—H12	74.7
O3—K2—S1	176.98 (7)	C14—C13—C12	115.2 (4)
N1—K2—S1	25.63 (7)	C14—C13—H13	122.4
S2—K2—S1	153.19 (4)	C12—C13—H13	122.4
C12^{iii}—K2—S1	67.56 (8)	N7—C14—C13	123.8 (5)
O3^{iii}—K2—K1^{i}	140.99 (7)	N7—C14—H14	118.1
O4W—K2—K1^{i}	81.88 (9)	C13—C14—H14	118.1
O3W—K2—K1^{i}	45.08 (7)	C20—C15—C16	120.1 (4)
O1—K2—K1^{i}	82.84 (7)	C20—C15—S2	120.3 (3)
N6^{iii}—K2—K1^{i}	153.33 (8)	C16—C15—S2	119.6 (3)
O4—K2—K1^{i}	39.76 (6)	C17—C16—C15	120.0 (4)
O3—K2—K1^{i}	86.84 (6)	C17—C16—H16	120.0
N1—K2—K1^{i}	94.45 (7)	C15—C16—H16	120.0
S2—K2—K1^{i}	61.95 (3)	C16—C17—C18	121.0 (4)
C12^{iii}—K2—K1^{i}	131.72 (8)	C16—C17—H17	119.5
S1—K2—K1^{i}	92.52 (3)	C18—C17—H17	119.5
O3^{iii}—K2—H5W	111.4 (10)	N8—C18—C19	120.3 (4)
O4W—K2—H5W	130.2 (11)	N8—C18—C17	121.9 (4)
O3W—K2—H5W	17.7 (2)	C19—C18—C17	117.7 (4)
O1—K2—H5W	65.1 (9)	C20—C19—C18	121.6 (4)
N6^{iii}—K2—H5W	128.6 (3)	C20—C19—H19	119.2
O4—K2—H5W	93.3 (2)	C18—C19—H19	119.2
O3—K2—H5W	123.2 (7)	C19—C20—C15	119.5 (4)
N1—K2—H5W	39.1 (3)	C19—C20—H20	120.2
S2—K2—H5W	102.5 (4)	C15—C20—H20	120.2
O2—S1—O1—K2	−107.96 (19)	C10—C5—C6—C7	0.0 (7)

695	N1—S1—O1—K2	18.6 (2)	S1—C5—C6—C7	176.6 (3)
696	C5—S1—O1—K2	132.83 (18)	C5—C6—C7—C8	−1.8 (7)
697	O4—S2—O3—K2^{iii}	−161.4 (2)	C6—C7—C8—C9	2.5 (7)
698	N5—S2—O3—K2^{iii}	−40.7 (3)	C6—C7—C8—N4	179.0 (4)
699	C15—S2—O3—K2^{iii}	81.7 (3)	C7—C8—C9—C10	−1.4 (7)
700	K2—S2—O3—K2^{iii}	−126.1 (2)	N4—C8—C9—C10	−178.0 (5)
701	O4—S2—O3—K2	−35.30 (18)	C8—C9—C10—C5	−0.3 (8)
702	N5—S2—O3—K2	85.46 (17)	C6—C5—C10—C9	1.0 (7)
703	C15—S2—O3—K2	−152.14 (15)	S1—C5—C10—C9	−175.6 (4)
704	O3—S2—O4—K1^{i}	143.5 (3)	C12—N6—C11—N7	−4.4 (6)
705	N5—S2—O4—K1^{i}	17.7 (3)	K2^{iii}—N6—C11—N7	121.7 (4)
706	C15—S2—O4—K1^{i}	−99.4 (3)	C12—N6—C11—N5	174.9 (4)
707	K2—S2—O4—K1^{i}	107.0 (3)	K2^{iii}—N6—C11—N5	−59.0 (5)
708	O3—S2—O4—K2	36.46 (19)	C12—N6—C11—K1	−88.0 (7)
709	N5—S2—O4—K2	−89.27 (17)	K2^{iii}—N6—C11—K1	38.2 (8)
710	C15—S2—O4—K2	153.58 (16)	C14—N7—C11—N6	4.7 (7)
711	O2—S1—N1—C1	−52.2 (4)	K1—N7—C11—N6	−148.8 (4)
712	O1—S1—N1—C1	−177.3 (3)	C14—N7—C11—N5	−174.6 (4)
713	C5—S1—N1—C1	69.1 (3)	K1—N7—C11—N5	32.0 (4)
714	K2—S1—N1—C1	−161.3 (4)	C14—N7—C11—K1	153.4 (5)
715	O2—S1—N1—K2	109.16 (17)	S2—N5—C11—N6	−3.2 (6)
716	O1—S1—N1—K2	−15.96 (17)	K1—N5—C11—N6	151.5 (4)
717	C5—S1—N1—K2	−129.60 (16)	S2—N5—C11—N7	176.2 (3)
718	O3—S2—N5—C11	54.6 (4)	K1—N5—C11—N7	−29.2 (4)
719	O4—S2—N5—C11	179.4 (3)	S2—N5—C11—K1	−154.7 (3)
720	C15—S2—N5—C11	−66.0 (4)	C11—N6—C12—C13	0.4 (6)
721	K2—S2—N5—C11	118.3 (3)	K2^{iii}—N6—C12—C13	−136.1 (4)
722	O3—S2—N5—K1	−85.3 (3)	C11—N6—C12—K2^{iii}	136.5 (4)
723	O4—S2—N5—K1	39.5 (3)	N6—C12—C13—C14	2.8 (7)
724	C15—S2—N5—K1	154.1 (2)	K2^{iii}—C12—C13—C14	−74.1 (7)
725	K2—S2—N5—K1	−21.6 (3)	C11—N7—C14—C13	−1.0 (7)
726	C2—N2—C1—N3	4.1 (6)	K1—N7—C14—C13	140.9 (4)
727	C2—N2—C1—N1	−175.5 (4)	C12—C13—C14—N7	−2.5 (7)
728	C4—N3—C1—N2	−4.1 (6)	O3—S2—C15—C20	−20.6 (4)
729	C4—N3—C1—N1	175.5 (4)	O4—S2—C15—C20	−141.6 (3)
730	S1—N1—C1—N2	175.3 (3)	N5—S2—C15—C20	104.4 (4)
731	K2—N1—C1—N2	25.0 (6)	K2—S2—C15—C20	−85.0 (4)
732	S1—N1—C1—N3	−4.4 (5)	O3—S2—C15—C16	160.5 (3)
733	K2—N1—C1—N3	−154.7 (3)	O4—S2—C15—C16	39.5 (4)
734	C1—N2—C2—C3	0.0 (7)	N5—S2—C15—C16	−74.4 (4)
735	N2—C2—C3—C4	−3.6 (7)	K2—S2—C15—C16	96.1 (4)
736	C1—N3—C4—C3	−0.1 (6)	C20—C15—C16—C17	0.4 (6)
737	C2—C3—C4—N3	3.7 (7)	S2—C15—C16—C17	179.2 (3)
738	O2—S1—C5—C6	143.5 (4)	C15—C16—C17—C18	1.4 (7)
739	O1—S1—C5—C6	−94.0 (4)	C16—C17—C18—N8	176.3 (4)
740	N1—S1—C5—C6	17.7 (4)	C16—C17—C18—C19	−1.8 (7)
741	K2—S1—C5—C6	−46.4 (4)	N8—C18—C19—C20	−177.7 (4)
742	O2—S1—C5—C10	−39.9 (4)	C17—C18—C19—C20	0.5 (6)

O1—S1—C5—C10	82.6 (4)	C18—C19—C20—C15	1.3 (6)
N1—S1—C5—C10	−165.7 (4)	C16—C15—C20—C19	−1.7 (6)
K2—S1—C5—C10	130.1 (3)	S2—C15—C20—C19	179.4 (3)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···O1^{i}	0.88 (1)	1.93 (2)	2.799 (5)	167 (8)
O1*W*—H2*W*···O4*W*^{i}	0.88 (1)	2.27 (4)	3.070 (6)	151 (6)
O2*W*—H3*W*···O3^{iii}	0.88 (1)	2.07 (2)	2.933 (5)	167 (6)
O2*W*—H4*W*···N2	0.88 (1)	1.97 (2)	2.828 (5)	167 (6)
O3*W*—H5*W*···N1	0.87 (1)	2.02 (2)	2.872 (5)	164 (5)
O3*W*—H6*W*···N5^{i}	0.87 (1)	1.98 (1)	2.835 (5)	166 (4)
O4*W*—H7*W*···O2*W*^{iv}	0.88 (1)	2.05 (2)	2.919 (6)	169 (7)
O4*W*—H8*W*···O4*W*^{v}	0.88 (1)	2.05 (2)	2.920 (8)	168 (9)
N4—H1*N*···N4^{vi}	0.87 (7)	2.50 (6)	3.054 (9)	122 (5)
N4—H2*N*···O2^{vii}	0.93 (7)	2.57 (7)	3.431 (7)	153 (5)
N8—H3*N*···O2^{viii}	0.87 (5)	2.38 (5)	3.046 (5)	134 (4)
N8—H3*N*···N3^{viii}	0.87 (5)	2.61 (5)	3.283 (6)	135 (4)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (iii) −*x*, −*y*+2, −*z*+1; (iv) *x*−1, *y*, *z*; (v) −*x*−1, −*y*+2, −*z*+1; (vi) −*x*−1, −*y*, −*z*; (vii) *x*, *y*−1, *z*; (viii) *x*, *y*, *z*+1.

(IV)

Crystal data

$C_{26}H_{32}N_6NaO_{14.50}S_3$
M_r = 779.74
Monoclinic, $P2_1/c$
a = 12.7688 (1) Å
b = 16.9040 (1) Å
c = 15.7411 (1) Å
β = 107.609 (1)°
V = 3238.42 (4) Å^3
Z = 4
F(000) = 1620
D_x = 1.599 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 35404 reflections
θ = 3.6–70.3°
μ = 2.95 mm^{-1}
T = 100 K
Block, red
0.12 × 0.08 × 0.04 mm

Data collection

XtaLAB AFC11 (RCD3) diffractometer
Radiation source: rotating anode
ω scans
Absorption correction: multi-scan *CrysAlis PRO* 1.171.39.34b (Rigaku Oxford Diffraction, 2017) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min} = 0.615, T_{max} = 1.000
59125 measured reflections
5913 independent reflections
5686 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 68.3°, θ_{min} = 3.6°
h = −15→15
k = −20→20
l = −18→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.075
S = 1.05
5913 reflections

537 parameters
13 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 2.347P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Na1	−0.36434 (5)	0.35305 (4)	0.00412 (4)	0.02103 (14)	
S1	0.25698 (3)	0.53159 (2)	0.56811 (2)	0.01894 (9)	
S2	0.28977 (3)	0.35439 (2)	0.12953 (2)	0.01723 (9)	
S3	0.34948 (3)	0.66079 (2)	0.22267 (2)	0.01823 (9)	
O1	0.30756 (9)	0.60882 (7)	0.58033 (7)	0.0231 (2)	
O2	0.25734 (9)	0.48924 (7)	0.64777 (7)	0.0232 (2)	
O1W	−0.35372 (9)	0.22020 (7)	0.04238 (8)	0.0218 (2)	
O3	−0.16759 (8)	0.36126 (6)	0.04200 (7)	0.0209 (2)	
O2W	−0.39276 (13)	0.36471 (9)	0.15242 (9)	0.0366 (3)	
O4	0.23184 (9)	0.28982 (6)	0.15504 (8)	0.0242 (2)	
O3W	−0.56243 (11)	0.33362 (8)	−0.06717 (10)	0.0324 (3)	
O5	0.29466 (9)	0.35183 (7)	0.03839 (7)	0.0247 (3)	
O4W	−0.40375 (13)	0.48626 (9)	−0.02427 (12)	0.0467 (4)	
H7W	−0.3673 (17)	0.5308 (9)	−0.0208 (17)	0.056*	
H8W	−0.449 (3)	0.501 (2)	0.006 (3)	0.056*	0.5
H14W	−0.463 (2)	0.499 (2)	−0.068 (2)	0.056*	0.5
O5W	−0.6489 (2)	0.33037 (15)	−0.24880 (18)	0.0323 (5)	0.5
H9W	−0.677 (3)	0.2873 (16)	−0.279 (2)	0.039*	0.5
H10W	−0.590 (2)	0.336 (2)	−0.267 (3)	0.039*	0.5
O6W	−0.3558 (3)	0.52405 (18)	0.16139 (18)	0.0459 (8)	0.5
H11W	−0.366 (2)	0.5317 (15)	0.2141 (10)	0.055*	
H12W	−0.367 (4)	0.4725 (8)	0.160 (3)	0.055*	0.5
O7W	−0.4311 (2)	0.54771 (18)	0.19266 (18)	0.0392 (7)	0.5
H13W	−0.451 (3)	0.566 (3)	0.2374 (18)	0.047*	0.5
O6	0.40067 (8)	0.36455 (6)	0.19268 (7)	0.0200 (2)	
O7	0.36235 (9)	0.69334 (7)	0.14107 (7)	0.0228 (2)	
O8	0.44918 (9)	0.62573 (7)	0.28078 (8)	0.0297 (3)	
O9	0.29864 (9)	0.71689 (7)	0.26880 (7)	0.0241 (2)	
N1	0.13018 (10)	0.53245 (8)	0.50886 (8)	0.0193 (3)	
N2	0.14382 (11)	0.63967 (8)	0.41387 (9)	0.0205 (3)	
N3	−0.02496 (10)	0.58329 (8)	0.41174 (9)	0.0202 (3)	
N4	0.47872 (12)	0.31848 (9)	0.37342 (10)	0.0228 (3)	
N5	0.04786 (10)	0.34343 (7)	0.02685 (8)	0.0165 (3)	
N6	−0.02618 (10)	0.29254 (7)	−0.01560 (8)	0.0173 (3)	

C1	0.08718 (12)	0.58593 (9)	0.44525 (10)	0.0185 (3)
C2	0.08701 (13)	0.69124 (10)	0.35332 (10)	0.0216 (3)
H2	0.1270	0.7293	0.3313	0.026*
C3	−0.02726 (13)	0.69278 (10)	0.32031 (11)	0.0232 (3)
H3	−0.0650	0.7314	0.2784	0.028*
C4	−0.08209 (13)	0.63587 (10)	0.35134 (11)	0.0238 (3)
H4	−0.1600	0.6334	0.3304	0.029*
C5	0.32606 (12)	0.47095 (9)	0.51081 (10)	0.0197 (3)
C6	0.35300 (12)	0.49962 (10)	0.43696 (10)	0.0223 (3)
H6	0.3365	0.5527	0.4176	0.027*
C7	0.40425 (12)	0.44943 (10)	0.39231 (10)	0.0219 (3)
H7	0.4236	0.4677	0.3420	0.026*
C8	0.42703 (12)	0.37216 (10)	0.42193 (10)	0.0205 (3)
C9	0.40333 (13)	0.34437 (10)	0.49661 (11)	0.0221 (3)
H9	0.4223	0.2919	0.5171	0.027*
C10	0.35154 (12)	0.39416 (10)	0.54122 (10)	0.0218 (3)
H10	0.3336	0.3759	0.5922	0.026*
C11	0.01948 (12)	0.40012 (9)	0.07444 (10)	0.0159 (3)
C12	−0.09441 (12)	0.41261 (9)	0.07379 (9)	0.0175 (3)
C13	−0.12211 (12)	0.48735 (9)	0.10541 (10)	0.0198 (3)
H13	−0.1955	0.4971	0.1051	0.024*
C14	−0.04523 (12)	0.54336 (9)	0.13538 (10)	0.0188 (3)
H14	−0.0671	0.5934	0.1516	0.023*
C15	0.06970 (12)	0.53059 (9)	0.14404 (10)	0.0166 (3)
C16	0.10495 (12)	0.45694 (9)	0.11881 (9)	0.0157 (3)
C17	0.22061 (12)	0.44559 (9)	0.13890 (10)	0.0168 (3)
C18	0.29200 (12)	0.50782 (9)	0.17202 (10)	0.0186 (3)
H18	0.3687	0.4999	0.1835	0.022*
C19	0.25427 (12)	0.58168 (9)	0.18898 (10)	0.0178 (3)
C20	0.14397 (12)	0.59221 (9)	0.17777 (10)	0.0178 (3)
H20	0.1182	0.6413	0.1929	0.021*
C21	−0.00296 (12)	0.23542 (9)	−0.07255 (10)	0.0178 (3)
C22	−0.09205 (13)	0.19251 (9)	−0.12561 (10)	0.0204 (3)
H22	−0.1643	0.2031	−0.1234	0.024*
C23	−0.07414 (14)	0.13423 (10)	−0.18169 (11)	0.0237 (3)
H23	−0.1343	0.1047	−0.2182	0.028*
C24	0.03150 (14)	0.11899 (10)	−0.18464 (11)	0.0243 (3)
H24	0.0438	0.0786	−0.2225	0.029*
C25	0.11952 (14)	0.16297 (10)	−0.13190 (11)	0.0242 (3)
H25	0.1916	0.1526	−0.1346	0.029*
C26	0.10358 (13)	0.22174 (10)	−0.07551 (10)	0.0211 (3)
H26	0.1637	0.2519	−0.0398	0.025*
H1W	−0.349 (2)	0.2133 (14)	0.0986 (18)	0.049 (7)*
H2W	−0.299 (2)	0.1888 (15)	0.0354 (16)	0.052 (7)*
H3W	−0.360 (2)	0.3264 (17)	0.1861 (18)	0.057 (8)*
H4W	−0.461 (3)	0.3655 (19)	0.161 (2)	0.089 (11)*
H5W	−0.607 (2)	0.3395 (16)	−0.0361 (19)	0.061 (8)*
H6W	−0.591 (3)	0.365 (2)	−0.109 (2)	0.086 (11)*

H1N	−0.0943 (19)	0.2989 (13)	−0.0086 (14)	0.039 (6)*
H2N	0.4668 (19)	0.2671 (15)	0.3875 (16)	0.046 (6)*
H3N	0.4510 (18)	0.3275 (13)	0.3137 (16)	0.036 (6)*
H4N	0.552 (2)	0.3289 (13)	0.3887 (14)	0.036 (6)*
H5N	−0.0568 (18)	0.5484 (13)	0.4332 (14)	0.034 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0167 (3)	0.0247 (3)	0.0222 (3)	0.0009 (2)	0.0066 (2)	0.0023 (2)
S1	0.01371 (18)	0.0258 (2)	0.01688 (18)	0.00205 (14)	0.00401 (14)	0.00107 (14)
S2	0.01208 (17)	0.02132 (19)	0.01829 (19)	0.00029 (13)	0.00458 (14)	0.00044 (14)
S3	0.01672 (18)	0.02092 (19)	0.01605 (18)	−0.00479 (14)	0.00343 (14)	−0.00034 (14)
O1	0.0168 (5)	0.0278 (6)	0.0235 (6)	−0.0008 (4)	0.0044 (4)	−0.0022 (5)
O2	0.0188 (5)	0.0335 (6)	0.0174 (5)	0.0043 (5)	0.0054 (4)	0.0033 (5)
O1W	0.0204 (6)	0.0252 (6)	0.0199 (6)	0.0006 (5)	0.0061 (4)	0.0006 (5)
O3	0.0146 (5)	0.0233 (6)	0.0249 (6)	−0.0033 (4)	0.0061 (4)	−0.0030 (4)
O2W	0.0409 (8)	0.0400 (8)	0.0350 (7)	0.0161 (6)	0.0208 (6)	0.0089 (6)
O4	0.0180 (5)	0.0200 (5)	0.0352 (6)	−0.0002 (4)	0.0090 (5)	0.0039 (5)
O3W	0.0246 (6)	0.0369 (7)	0.0407 (8)	0.0063 (5)	0.0171 (6)	0.0062 (6)
O5	0.0170 (5)	0.0378 (7)	0.0201 (6)	0.0016 (5)	0.0069 (4)	−0.0037 (5)
O4W	0.0522 (9)	0.0349 (7)	0.0683 (11)	0.0142 (7)	0.0413 (8)	0.0217 (7)
O5W	0.0279 (13)	0.0334 (14)	0.0360 (14)	−0.0018 (10)	0.0103 (11)	−0.0034 (11)
O6W	0.070 (2)	0.0441 (16)	0.0218 (13)	0.0273 (15)	0.0117 (13)	0.0043 (12)
O7W	0.0281 (14)	0.0483 (17)	0.0362 (15)	0.0008 (12)	0.0021 (11)	0.0159 (13)
O6	0.0129 (5)	0.0250 (6)	0.0204 (5)	0.0012 (4)	0.0026 (4)	0.0015 (4)
O7	0.0257 (6)	0.0247 (6)	0.0198 (6)	−0.0074 (5)	0.0098 (5)	−0.0014 (4)
O8	0.0205 (6)	0.0305 (6)	0.0299 (6)	−0.0045 (5)	−0.0047 (5)	0.0029 (5)
O9	0.0250 (6)	0.0269 (6)	0.0223 (6)	−0.0072 (5)	0.0101 (5)	−0.0067 (5)
N1	0.0130 (6)	0.0250 (7)	0.0192 (6)	0.0018 (5)	0.0039 (5)	0.0018 (5)
N2	0.0174 (6)	0.0246 (7)	0.0192 (6)	0.0006 (5)	0.0050 (5)	0.0011 (5)
N3	0.0142 (6)	0.0266 (7)	0.0201 (7)	0.0013 (5)	0.0058 (5)	0.0025 (5)
N4	0.0166 (7)	0.0317 (8)	0.0209 (7)	0.0058 (6)	0.0066 (6)	0.0019 (6)
N5	0.0158 (6)	0.0176 (6)	0.0149 (6)	−0.0007 (5)	0.0028 (5)	0.0023 (5)
N6	0.0139 (6)	0.0202 (6)	0.0172 (6)	−0.0010 (5)	0.0038 (5)	−0.0005 (5)
C1	0.0158 (7)	0.0226 (8)	0.0173 (7)	0.0023 (6)	0.0053 (6)	−0.0031 (6)
C2	0.0233 (8)	0.0234 (8)	0.0191 (8)	0.0006 (6)	0.0078 (6)	−0.0010 (6)
C3	0.0225 (8)	0.0274 (8)	0.0193 (8)	0.0058 (7)	0.0057 (6)	0.0030 (6)
C4	0.0168 (8)	0.0331 (9)	0.0202 (8)	0.0056 (6)	0.0040 (6)	0.0021 (7)
C5	0.0114 (7)	0.0282 (8)	0.0183 (7)	0.0010 (6)	0.0028 (6)	0.0003 (6)
C6	0.0180 (7)	0.0275 (8)	0.0202 (8)	0.0030 (6)	0.0042 (6)	0.0036 (6)
C7	0.0163 (7)	0.0316 (9)	0.0177 (7)	0.0027 (6)	0.0050 (6)	0.0039 (6)
C8	0.0115 (7)	0.0304 (9)	0.0186 (8)	0.0032 (6)	0.0032 (6)	0.0002 (6)
C9	0.0174 (7)	0.0271 (8)	0.0214 (8)	0.0041 (6)	0.0052 (6)	0.0039 (6)
C10	0.0159 (7)	0.0304 (9)	0.0190 (8)	0.0014 (6)	0.0050 (6)	0.0035 (6)
C11	0.0144 (7)	0.0181 (7)	0.0145 (7)	0.0001 (6)	0.0036 (5)	0.0024 (6)
C12	0.0155 (7)	0.0224 (8)	0.0140 (7)	−0.0009 (6)	0.0035 (6)	0.0024 (6)
C13	0.0150 (7)	0.0251 (8)	0.0203 (8)	0.0015 (6)	0.0069 (6)	0.0004 (6)

C14	0.0189 (7)	0.0205 (8)	0.0179 (7)	0.0021 (6)	0.0067 (6)	0.0009 (6)
C15	0.0165 (7)	0.0205 (7)	0.0125 (7)	−0.0002 (6)	0.0041 (6)	0.0030 (6)
C16	0.0153 (7)	0.0202 (7)	0.0117 (7)	−0.0006 (6)	0.0045 (6)	0.0033 (6)
C17	0.0157 (7)	0.0207 (7)	0.0141 (7)	−0.0002 (6)	0.0048 (6)	0.0026 (6)
C18	0.0148 (7)	0.0240 (8)	0.0161 (7)	−0.0016 (6)	0.0034 (6)	0.0037 (6)
C19	0.0175 (7)	0.0212 (7)	0.0131 (7)	−0.0032 (6)	0.0024 (6)	0.0029 (6)
C20	0.0199 (7)	0.0189 (7)	0.0147 (7)	−0.0010 (6)	0.0051 (6)	0.0014 (6)
C21	0.0201 (7)	0.0175 (7)	0.0155 (7)	0.0010 (6)	0.0049 (6)	0.0021 (6)
C22	0.0189 (7)	0.0225 (8)	0.0196 (8)	−0.0017 (6)	0.0055 (6)	0.0016 (6)
C23	0.0256 (8)	0.0234 (8)	0.0209 (8)	−0.0058 (6)	0.0051 (7)	−0.0024 (6)
C24	0.0312 (9)	0.0229 (8)	0.0187 (8)	0.0022 (7)	0.0077 (7)	−0.0019 (6)
C25	0.0208 (8)	0.0294 (9)	0.0225 (8)	0.0046 (7)	0.0065 (6)	−0.0006 (7)
C26	0.0179 (7)	0.0247 (8)	0.0193 (8)	0.0003 (6)	0.0034 (6)	−0.0009 (6)

Geometric parameters (Å, º)

Na1—O1W	2.3184 (13)	N5—C11	1.3322 (19)
Na1—O4W	2.3211 (15)	N6—C21	1.409 (2)
Na1—O3	2.4042 (12)	N6—H1N	0.92 (2)
Na1—O7^{i}	2.4237 (12)	C2—C3	1.393 (2)
Na1—O3W	2.4592 (14)	C2—H2	0.9500
Na1—O2W	2.4764 (14)	C3—C4	1.364 (2)
S1—O2	1.4428 (11)	C3—H3	0.9500
S1—O1	1.4433 (12)	C4—H4	0.9500
S1—N1	1.6055 (13)	C5—C10	1.388 (2)
S1—C5	1.7674 (16)	C5—C6	1.395 (2)
S2—O4	1.4419 (11)	C6—C7	1.386 (2)
S2—O5	1.4554 (12)	C6—H6	0.9500
S2—O6	1.4735 (11)	C7—C8	1.388 (2)
S2—C17	1.8048 (15)	C7—H7	0.9500
S3—O8	1.4506 (12)	C8—C9	1.382 (2)
S3—O7	1.4516 (11)	C9—C10	1.385 (2)
S3—O9	1.4605 (12)	C9—H9	0.9500
S3—C19	1.7769 (15)	C10—H10	0.9500
O1W—H1W	0.88 (3)	C11—C16	1.463 (2)
O1W—H2W	0.91 (3)	C11—C12	1.467 (2)
O3—C12	1.2626 (18)	C12—C13	1.441 (2)
O2W—H3W	0.86 (3)	C13—C14	1.343 (2)
O2W—H4W	0.92 (4)	C13—H13	0.9500
O3W—H5W	0.86 (3)	C14—C15	1.448 (2)
O3W—H6W	0.83 (4)	C14—H14	0.9500
O4W—H7W	0.879 (10)	C15—C20	1.401 (2)
O4W—H8W	0.883 (10)	C15—C16	1.421 (2)
O4W—H14W	0.886 (10)	C16—C17	1.427 (2)
O5W—H9W	0.883 (10)	C17—C18	1.386 (2)
O5W—H10W	0.885 (10)	C18—C19	1.393 (2)
O6W—H11W	0.886 (10)	C18—H18	0.9500
O6W—H12W	0.881 (10)	C19—C20	1.377 (2)

O7W—H13W	0.875 (10)	C20—H20	0.9500
O7—Na1^{i}	2.4237 (12)	C21—C22	1.394 (2)
N1—C1	1.337 (2)	C21—C26	1.395 (2)
N2—C2	1.333 (2)	C22—C23	1.387 (2)
N2—C1	1.345 (2)	C22—H22	0.9500
N3—C4	1.344 (2)	C23—C24	1.388 (2)
N3—C1	1.369 (2)	C23—H23	0.9500
N3—H5N	0.84 (2)	C24—C25	1.393 (2)
N4—C8	1.465 (2)	C24—H24	0.9500
N4—H2N	0.92 (3)	C25—C26	1.388 (2)
N4—H3N	0.91 (2)	C25—H25	0.9500
N4—H4N	0.91 (2)	C26—H26	0.9500
N5—N6	1.3031 (18)		
O1W—Na1—O4W	169.11 (5)	C4—C3—C2	116.51 (15)
O1W—Na1—O3	90.96 (4)	C4—C3—H3	121.7
O4W—Na1—O3	98.12 (5)	C2—C3—H3	121.7
O1W—Na1—O7^{i}	85.13 (4)	N3—C4—C3	119.48 (15)
O4W—Na1—O7^{i}	101.30 (5)	N3—C4—H4	120.3
O3—Na1—O7^{i}	86.81 (4)	C3—C4—H4	120.3
O1W—Na1—O3W	87.73 (5)	C10—C5—C6	121.41 (15)
O4W—Na1—O3W	84.75 (6)	C10—C5—S1	117.89 (12)
O3—Na1—O3W	166.97 (5)	C6—C5—S1	120.71 (12)
O7^{i}—Na1—O3W	80.17 (5)	C7—C6—C5	118.90 (15)
O1W—Na1—O2W	81.14 (5)	C7—C6—H6	120.6
O4W—Na1—O2W	91.10 (6)	C5—C6—H6	120.6
O3—Na1—O2W	101.64 (5)	C6—C7—C8	119.27 (15)
O7^{i}—Na1—O2W	163.94 (5)	C6—C7—H7	120.4
O3W—Na1—O2W	90.97 (5)	C8—C7—H7	120.4
O2—S1—O1	116.71 (7)	C9—C8—C7	121.88 (15)
O2—S1—N1	104.29 (7)	C9—C8—N4	118.81 (15)
O1—S1—N1	113.65 (7)	C7—C8—N4	119.30 (14)
O2—S1—C5	106.55 (7)	C8—C9—C10	119.06 (15)
O1—S1—C5	108.68 (7)	C8—C9—H9	120.5
N1—S1—C5	106.26 (7)	C10—C9—H9	120.5
O4—S2—O5	115.51 (7)	C9—C10—C5	119.43 (15)
O4—S2—O6	111.69 (7)	C9—C10—H10	120.3
O5—S2—O6	110.45 (6)	C5—C10—H10	120.3
O4—S2—C17	108.60 (7)	N5—C11—C16	116.76 (13)
O5—S2—C17	105.90 (7)	N5—C11—C12	122.51 (13)
O6—S2—C17	103.85 (7)	C16—C11—C12	120.25 (13)
O8—S3—O7	113.59 (7)	O3—C12—C13	120.68 (13)
O8—S3—O9	112.90 (7)	O3—C12—C11	121.14 (14)
O7—S3—O9	112.34 (7)	C13—C12—C11	118.06 (13)
O8—S3—C19	105.75 (7)	C14—C13—C12	120.66 (14)
O7—S3—C19	105.76 (7)	C14—C13—H13	119.7
O9—S3—C19	105.67 (7)	C12—C13—H13	119.7
Na1—O1W—H1W	111.8 (16)	C13—C14—C15	122.71 (14)

Na1—O1W—H2W	121.3 (16)	C13—C14—H14	118.6
H1W—O1W—H2W	103 (2)	C15—C14—H14	118.6
C12—O3—Na1	136.93 (10)	C20—C15—C16	121.48 (13)
Na1—O2W—H3W	110.0 (18)	C20—C15—C14	118.39 (14)
Na1—O2W—H4W	124 (2)	C16—C15—C14	120.13 (13)
H3W—O2W—H4W	102 (3)	C15—C16—C17	116.75 (13)
Na1—O3W—H5W	119.2 (19)	C15—C16—C11	117.08 (13)
Na1—O3W—H6W	115 (2)	C17—C16—C11	126.18 (13)
H5W—O3W—H6W	100 (3)	C18—C17—C16	120.02 (14)
Na1—O4W—H7W	137.7 (16)	C18—C17—S2	113.36 (11)
Na1—O4W—H8W	108 (3)	C16—C17—S2	126.58 (11)
H7W—O4W—H8W	98 (2)	C17—C18—C19	121.74 (14)
Na1—O4W—H14W	118 (3)	C17—C18—H18	119.1
H7W—O4W—H14W	99 (2)	C19—C18—H18	119.1
H9W—O5W—H10W	99 (2)	C20—C19—C18	119.40 (14)
H11W—O6W—H12W	96 (2)	C20—C19—S3	121.46 (12)
S3—O7—Na1^{i}	138.42 (7)	C18—C19—S3	119.13 (11)
C1—N1—S1	123.67 (11)	C19—C20—C15	120.10 (14)
C2—N2—C1	117.89 (13)	C19—C20—H20	119.9
C4—N3—C1	122.12 (14)	C15—C20—H20	119.9
C4—N3—H5N	121.4 (15)	C22—C21—C26	121.42 (14)
C1—N3—H5N	116.4 (15)	C22—C21—N6	116.56 (13)
C8—N4—H2N	109.0 (15)	C26—C21—N6	122.01 (14)
C8—N4—H3N	109.8 (14)	C23—C22—C21	119.28 (15)
H2N—N4—H3N	111 (2)	C23—C22—H22	120.4
C8—N4—H4N	109.3 (14)	C21—C22—H22	120.4
H2N—N4—H4N	111 (2)	C22—C23—C24	120.15 (15)
H3N—N4—H4N	106.8 (19)	C22—C23—H23	119.9
N6—N5—C11	118.99 (13)	C24—C23—H23	119.9
N5—N6—C21	121.27 (13)	C23—C24—C25	119.87 (15)
N5—N6—H1N	115.4 (14)	C23—C24—H24	120.1
C21—N6—H1N	123.2 (14)	C25—C24—H24	120.1
N1—C1—N2	125.95 (14)	C26—C25—C24	121.02 (15)
N1—C1—N3	114.28 (14)	C26—C25—H25	119.5
N2—C1—N3	119.77 (14)	C24—C25—H25	119.5
N2—C2—C3	124.12 (15)	C25—C26—C21	118.25 (14)
N2—C2—H2	117.9	C25—C26—H26	120.9
C3—C2—H2	117.9	C21—C26—H26	120.9

O8—S3—O7—Na1^{i}	81.07 (11)	C13—C14—C15—C20	−178.66 (14)
O9—S3—O7—Na1^{i}	−149.26 (9)	C13—C14—C15—C16	1.2 (2)
C19—S3—O7—Na1^{i}	−34.46 (12)	C20—C15—C16—C17	6.9 (2)
O2—S1—N1—C1	156.92 (13)	C14—C15—C16—C17	−172.92 (13)
O1—S1—N1—C1	28.75 (15)	C20—C15—C16—C11	−172.79 (13)
C5—S1—N1—C1	−90.72 (14)	C14—C15—C16—C11	7.4 (2)
C11—N5—N6—C21	174.83 (13)	N5—C11—C16—C15	159.68 (13)
S1—N1—C1—N2	8.8 (2)	C12—C11—C16—C15	−12.5 (2)
S1—N1—C1—N3	−170.92 (11)	N5—C11—C16—C17	−20.0 (2)

C2—N2—C1—N1	−176.66 (15)	C12—C11—C16—C17	167.76 (14)
C2—N2—C1—N3	3.0 (2)	C15—C16—C17—C18	−7.1 (2)
C4—N3—C1—N1	175.88 (14)	C11—C16—C17—C18	172.62 (14)
C4—N3—C1—N2	−3.8 (2)	C15—C16—C17—S2	170.58 (11)
C1—N2—C2—C3	−0.2 (2)	C11—C16—C17—S2	−9.7 (2)
N2—C2—C3—C4	−1.9 (2)	O4—S2—C17—C18	141.14 (11)
C1—N3—C4—C3	1.6 (2)	O5—S2—C17—C18	−94.23 (12)
C2—C3—C4—N3	1.1 (2)	O6—S2—C17—C18	22.15 (13)
O2—S1—C5—C10	9.38 (14)	O4—S2—C17—C16	−36.66 (15)
O1—S1—C5—C10	135.92 (12)	O5—S2—C17—C16	87.97 (14)
N1—S1—C5—C10	−101.41 (13)	O6—S2—C17—C16	−155.65 (13)
O2—S1—C5—C6	−171.14 (12)	C16—C17—C18—C19	1.8 (2)
O1—S1—C5—C6	−44.60 (14)	S2—C17—C18—C19	−176.12 (11)
N1—S1—C5—C6	78.07 (14)	C17—C18—C19—C20	4.0 (2)
C10—C5—C6—C7	1.3 (2)	C17—C18—C19—S3	−174.87 (11)
S1—C5—C6—C7	−178.11 (12)	O8—S3—C19—C20	143.56 (13)
C5—C6—C7—C8	0.2 (2)	O7—S3—C19—C20	−95.67 (13)
C6—C7—C8—C9	−2.2 (2)	O9—S3—C19—C20	23.63 (14)
C6—C7—C8—N4	178.48 (14)	O8—S3—C19—C18	−37.62 (14)
C7—C8—C9—C10	2.5 (2)	O7—S3—C19—C18	83.15 (13)
N4—C8—C9—C10	−178.13 (14)	O9—S3—C19—C18	−157.55 (12)
C8—C9—C10—C5	−0.9 (2)	C18—C19—C20—C15	−4.2 (2)
C6—C5—C10—C9	−1.0 (2)	S3—C19—C20—C15	174.65 (11)
S1—C5—C10—C9	178.47 (12)	C16—C15—C20—C19	−1.4 (2)
N6—N5—C11—C16	−178.41 (12)	C14—C15—C20—C19	178.45 (13)
N6—N5—C11—C12	−6.4 (2)	N5—N6—C21—C22	−169.89 (13)
Na1—O3—C12—C13	6.3 (2)	N5—N6—C21—C26	10.6 (2)
Na1—O3—C12—C11	−169.80 (10)	C26—C21—C22—C23	0.9 (2)
N5—C11—C12—O3	13.8 (2)	N6—C21—C22—C23	−178.63 (14)
C16—C11—C12—O3	−174.40 (13)	C21—C22—C23—C24	0.1 (2)
N5—C11—C12—C13	−162.36 (14)	C22—C23—C24—C25	−0.7 (2)
C16—C11—C12—C13	9.4 (2)	C23—C24—C25—C26	0.5 (3)
O3—C12—C13—C14	−176.90 (14)	C24—C25—C26—C21	0.4 (2)
C11—C12—C13—C14	−0.7 (2)	C22—C21—C26—C25	−1.1 (2)
C12—C13—C14—C15	−4.7 (2)	N6—C21—C26—C25	178.40 (14)

Symmetry code: (i) −*x*, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···O9^{ii}	0.88 (3)	1.99 (3)	2.8404 (16)	163 (2)
O1*W*—H2*W*···O1^{ii}	0.91 (3)	2.24 (3)	2.8830 (17)	127 (2)
O1*W*—H2*W*···N2^{ii}	0.91 (3)	2.07 (3)	2.8972 (18)	150 (2)
O2*W*—H3*W*···O9^{ii}	0.86 (3)	2.05 (3)	2.8852 (18)	163 (2)
O2*W*—H4*W*···O6^{iii}	0.92 (4)	1.98 (4)	2.8932 (18)	174 (3)
O3*W*—H5*W*···O5^{iii}	0.86 (3)	1.98 (3)	2.8320 (18)	178 (3)
O3*W*—H6*W*···O5*W*	0.83 (4)	2.18 (3)	2.736 (3)	124 (3)

O3*W*—H6*W*···O6*W*^{iv}	0.83 (4)	2.09 (4)	2.853 (3)	153 (3)
O3*W*—H6*W*···O7*W*^{iv}	0.83 (4)	2.06 (4)	2.799 (3)	148 (3)
O4*W*—H7*W*···O5^{i}	0.88 (1)	2.24 (1)	3.1093 (19)	169 (2)
O4*W*—H8*W*···O4*W*^{iv}	0.88 (1)	2.00 (2)	2.825 (3)	154 (4)
O4*W*—H8*W*···O6*W*	0.88 (1)	2.41 (5)	2.871 (3)	113 (4)
O4*W*—H14*W*···O7*W*^{iv}	0.89 (1)	2.16 (3)	2.902 (3)	140 (4)
O5*W*—H9*W*···O4^{v}	0.88 (1)	1.85 (1)	2.710 (3)	166 (4)
O5*W*—H10*W*···O8^{i}	0.89 (1)	1.99 (1)	2.843 (3)	163 (3)
O6*W*—H11*W*···O2^{vi}	0.89 (1)	2.22 (2)	2.893 (3)	132 (2)
O6*W*—H12*W*···O2*W*	0.88 (1)	1.85 (1)	2.731 (3)	178 (5)
O7*W*—H13*W*···O8^{iii}	0.88 (1)	1.91 (2)	2.699 (3)	150 (3)
N6—H1*N*···O3	0.92 (2)	1.75 (2)	2.5342 (16)	142 (2)
N4—H2*N*···O3*W*^{vii}	0.92 (3)	1.93 (3)	2.839 (2)	171 (2)
N4—H3*N*···O6	0.91 (2)	1.92 (2)	2.8245 (18)	170 (2)
N4—H4*N*···O1^{viii}	0.91 (2)	2.01 (2)	2.8784 (18)	159.6 (19)
N4—H4*N*···O1*W*^{vii}	0.91 (2)	2.50 (2)	2.9386 (19)	110.5 (16)
N3—H5*N*···O2^{vi}	0.84 (2)	2.57 (2)	3.0827 (17)	120.3 (18)
N3—H5*N*···N1^{vi}	0.84 (2)	2.02 (2)	2.8655 (19)	177 (2)

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) −*x*, *y*−1/2, −*z*+1/2; (iii) *x*−1, *y*, *z*; (iv) −*x*−1, −*y*+1, −*z*; (v) *x*−1, −*y*+1/2, *z*−1/2; (vi) −*x*, −*y*+1, −*z*+1; (vii) *x*+1, −*y*+1/2, *z*+1/2; (viii) −*x*+1, −*y*+1, −*z*+1.

(V)

Crystal data

$C_{15}H_{16}N_6O_2S$
$M_r = 344.40$
Monoclinic, $P2_1/c$
$a = 8.5796$ (2) Å
$b = 19.0371$ (5) Å
$c = 11.2512$ (4) Å
$\beta = 121.116$ (3)°
$V = 1573.27$ (9) Å^3
$Z = 4$
$F(000) = 720$
$D_x = 1.454$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8493 reflections
$\theta = 3.0$–29.5°
$\mu = 0.23$ mm^{-1}
$T = 123$ K
Sheet, colourless
0.4 × 0.3 × 0.02 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: sealed tube
ω scans
Absorption correction: multi-scan *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
$T_{min} = 0.527$, $T_{max} = 1.000$
22193 measured reflections
4082 independent reflections
3225 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 29.0°$, $\theta_{min} = 3.0°$
$h = -11 \rightarrow 11$
$k = -25 \rightarrow 25$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.114$
$S = 1.04$
4082 reflections
237 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.8192P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.87444 (6)	0.84851 (2)	0.27777 (4)	0.02297 (12)
O1	0.97267 (17)	0.79251 (7)	0.25677 (14)	0.0309 (3)
O2	0.96376 (16)	0.87572 (7)	0.41782 (12)	0.0271 (3)
N1	0.83358 (19)	0.90544 (7)	0.16260 (15)	0.0233 (3)
N2	0.73473 (18)	1.01144 (7)	0.05571 (14)	0.0216 (3)
N3	0.71259 (19)	0.98683 (8)	0.25488 (15)	0.0239 (3)
N4	0.1507 (2)	0.73439 (9)	0.12033 (18)	0.0284 (3)
N5	1.20543 (19)	1.03376 (7)	0.15032 (15)	0.0231 (3)
N6	1.0916 (3)	1.14087 (10)	0.0502 (2)	0.0411 (4)
C1	0.7592 (2)	0.96881 (9)	0.16105 (17)	0.0209 (3)
C2	0.6662 (2)	1.07527 (9)	0.04854 (17)	0.0238 (4)
H2	0.6475	1.1057	−0.0247	0.029*
C3	0.6214 (2)	1.09897 (9)	0.14288 (18)	0.0258 (4)
H3	0.5754	1.1449	0.1382	0.031*
C4	0.6476 (2)	1.05165 (9)	0.24444 (18)	0.0258 (4)
H4	0.6175	1.0661	0.3110	0.031*
C5	0.6654 (2)	0.81161 (8)	0.23937 (17)	0.0215 (3)
C6	0.5823 (2)	0.76230 (9)	0.13303 (18)	0.0266 (4)
H6	0.6422	0.7470	0.0864	0.032*
C7	0.4129 (3)	0.73551 (9)	0.09498 (18)	0.0275 (4)
H7	0.3577	0.7015	0.0230	0.033*
C8	0.3216 (2)	0.75825 (8)	0.16202 (17)	0.0219 (3)
C9	0.4076 (2)	0.80709 (9)	0.26908 (17)	0.0235 (3)
H9	0.3488	0.8224	0.3164	0.028*
C10	0.5776 (2)	0.83359 (9)	0.30745 (17)	0.0235 (3)
H10	0.6342	0.8669	0.3806	0.028*
C11	1.1346 (2)	1.09647 (9)	0.15389 (18)	0.0251 (4)
C12	1.1082 (2)	1.10992 (10)	0.2655 (2)	0.0316 (4)
H12	1.0611	1.1540	0.2721	0.038*
C13	1.1499 (3)	1.06005 (11)	0.3633 (2)	0.0340 (4)

H13	1.1283	1.0688	0.4367	0.041*
C14	1.2247 (3)	0.99557 (11)	0.3572 (2)	0.0368 (5)
H14	1.2565	0.9607	0.4264	0.044*
C15	1.2502 (3)	0.98455 (10)	0.2496 (2)	0.0313 (4)
H15	1.3008	0.9412	0.2437	0.038*
H1N	0.109 (3)	0.6980 (12)	0.067 (2)	0.036 (6)*
H2N	0.100 (3)	0.7493 (12)	0.165 (2)	0.038 (6)*
H3N	1.220 (3)	1.0215 (12)	0.078 (2)	0.044 (6)*
H4N	1.045 (3)	1.1818 (14)	0.050 (3)	0.051 (7)*
H5N	1.105 (4)	1.1256 (13)	−0.021 (3)	0.060 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0214 (2)	0.0248 (2)	0.0239 (2)	0.00462 (16)	0.01254 (17)	0.00161 (16)
O1	0.0288 (7)	0.0315 (7)	0.0364 (7)	0.0096 (5)	0.0197 (6)	0.0023 (6)
O2	0.0236 (6)	0.0309 (7)	0.0230 (6)	0.0037 (5)	0.0092 (5)	0.0021 (5)
N1	0.0221 (7)	0.0251 (7)	0.0242 (7)	0.0019 (6)	0.0130 (6)	0.0013 (6)
N2	0.0188 (7)	0.0236 (7)	0.0215 (7)	−0.0033 (5)	0.0097 (6)	−0.0017 (5)
N3	0.0220 (7)	0.0278 (7)	0.0224 (7)	0.0011 (6)	0.0119 (6)	0.0000 (6)
N4	0.0302 (8)	0.0267 (8)	0.0333 (9)	−0.0041 (7)	0.0201 (7)	−0.0066 (7)
N5	0.0230 (7)	0.0257 (7)	0.0230 (7)	0.0009 (6)	0.0135 (6)	−0.0012 (6)
N6	0.0551 (12)	0.0302 (9)	0.0434 (11)	0.0162 (8)	0.0292 (10)	0.0084 (8)
C1	0.0161 (7)	0.0247 (8)	0.0203 (8)	−0.0027 (6)	0.0082 (6)	−0.0017 (6)
C2	0.0219 (8)	0.0241 (8)	0.0222 (8)	−0.0031 (7)	0.0092 (7)	0.0001 (7)
C3	0.0235 (8)	0.0219 (8)	0.0267 (9)	−0.0007 (7)	0.0092 (7)	−0.0045 (7)
C4	0.0215 (8)	0.0300 (9)	0.0247 (8)	−0.0001 (7)	0.0111 (7)	−0.0047 (7)
C5	0.0238 (8)	0.0199 (8)	0.0227 (8)	0.0031 (6)	0.0135 (7)	0.0024 (6)
C6	0.0310 (9)	0.0265 (8)	0.0288 (9)	0.0033 (7)	0.0201 (8)	−0.0030 (7)
C7	0.0322 (9)	0.0250 (8)	0.0270 (9)	−0.0013 (7)	0.0166 (8)	−0.0071 (7)
C8	0.0261 (8)	0.0183 (7)	0.0232 (8)	0.0027 (6)	0.0141 (7)	0.0044 (6)
C9	0.0304 (9)	0.0229 (8)	0.0234 (8)	0.0022 (7)	0.0183 (7)	0.0015 (7)
C10	0.0293 (9)	0.0228 (8)	0.0203 (8)	0.0014 (7)	0.0142 (7)	−0.0009 (6)
C11	0.0195 (8)	0.0253 (8)	0.0286 (9)	0.0006 (7)	0.0111 (7)	−0.0029 (7)
C12	0.0248 (9)	0.0353 (10)	0.0367 (10)	−0.0030 (8)	0.0173 (8)	−0.0133 (8)
C13	0.0268 (9)	0.0514 (12)	0.0281 (9)	−0.0147 (9)	0.0173 (8)	−0.0168 (9)
C14	0.0405 (11)	0.0416 (11)	0.0289 (10)	−0.0055 (9)	0.0185 (9)	0.0039 (8)
C15	0.0368 (10)	0.0270 (9)	0.0331 (10)	0.0041 (8)	0.0202 (9)	0.0033 (8)

Geometric parameters (Å, º)

S1—O2	1.4458 (13)	C3—H3	0.9500
S1—O1	1.4520 (12)	C4—H4	0.9500
S1—N1	1.5834 (15)	C5—C10	1.388 (2)
S1—C5	1.7592 (17)	C5—C6	1.393 (2)
N1—C1	1.361 (2)	C6—C7	1.383 (3)
N2—C2	1.334 (2)	C6—H6	0.9500
N2—C1	1.360 (2)	C7—C8	1.407 (2)

N3—C4	1.334 (2)	C7—H7	0.9500
N3—C1	1.352 (2)	C8—C9	1.393 (2)
N4—C8	1.366 (2)	C9—C10	1.384 (2)
N4—H1N	0.86 (2)	C9—H9	0.9500
N4—H2N	0.87 (2)	C10—H10	0.9500
N5—C11	1.349 (2)	C11—C12	1.410 (3)
N5—C15	1.353 (2)	C12—C13	1.354 (3)
N5—H3N	0.92 (2)	C12—H12	0.9500
N6—C11	1.329 (3)	C13—C14	1.403 (3)
N6—H4N	0.87 (3)	C13—H13	0.9500
N6—H5N	0.92 (3)	C14—C15	1.352 (3)
C2—C3	1.379 (2)	C14—H14	0.9500
C2—H2	0.9500	C15—H15	0.9500
C3—C4	1.379 (3)		

O2—S1—O1	114.52 (8)	C6—C5—S1	118.99 (13)
O2—S1—N1	114.53 (8)	C7—C6—C5	120.28 (16)
O1—S1—N1	105.72 (8)	C7—C6—H6	119.9
O2—S1—C5	108.04 (8)	C5—C6—H6	119.9
O1—S1—C5	105.70 (8)	C6—C7—C8	120.50 (16)
N1—S1—C5	107.81 (8)	C6—C7—H7	119.7
C1—N1—S1	121.43 (12)	C8—C7—H7	119.7
C2—N2—C1	117.44 (14)	N4—C8—C9	120.93 (16)
C4—N3—C1	116.41 (15)	N4—C8—C7	120.64 (16)
C8—N4—H1N	119.2 (15)	C9—C8—C7	118.40 (16)
C8—N4—H2N	117.6 (15)	C10—C9—C8	121.00 (16)
H1N—N4—H2N	121 (2)	C10—C9—H9	119.5
C11—N5—C15	121.88 (16)	C8—C9—H9	119.5
C11—N5—H3N	121.5 (14)	C9—C10—C5	120.19 (16)
C15—N5—H3N	116.5 (14)	C9—C10—H10	119.9
C11—N6—H4N	119.3 (16)	C5—C10—H10	119.9
C11—N6—H5N	117.6 (16)	N6—C11—N5	117.51 (17)
H4N—N6—H5N	123 (2)	N6—C11—C12	124.49 (18)
N3—C1—N2	123.66 (15)	N5—C11—C12	117.99 (17)
N3—C1—N1	122.74 (15)	C13—C12—C11	119.97 (17)
N2—C1—N1	113.60 (14)	C13—C12—H12	120.0
N2—C2—C3	122.76 (16)	C11—C12—H12	120.0
N2—C2—H2	118.6	C12—C13—C14	120.61 (17)
C3—C2—H2	118.6	C12—C13—H13	119.7
C2—C3—C4	115.67 (16)	C14—C13—H13	119.7
C2—C3—H3	122.2	C15—C14—C13	117.98 (18)
C4—C3—H3	122.2	C15—C14—H14	121.0
N3—C4—C3	123.98 (16)	C13—C14—H14	121.0
N3—C4—H4	118.0	C14—C15—N5	121.55 (18)
C3—C4—H4	118.0	C14—C15—H15	119.2
C10—C5—C6	119.61 (16)	N5—C15—H15	119.2
C10—C5—S1	121.30 (13)		

O2—S1—N1—C1	−46.01 (15)	C10—C5—C6—C7	0.2 (3)
O1—S1—N1—C1	−173.05 (13)	S1—C5—C6—C7	−176.15 (14)
C5—S1—N1—C1	74.26 (14)	C5—C6—C7—C8	0.7 (3)
C4—N3—C1—N2	−3.3 (2)	C6—C7—C8—N4	176.89 (16)
C4—N3—C1—N1	177.47 (15)	C6—C7—C8—C9	−1.3 (3)
C2—N2—C1—N3	2.2 (2)	N4—C8—C9—C10	−177.19 (16)
C2—N2—C1—N1	−178.51 (14)	C7—C8—C9—C10	1.0 (2)
S1—N1—C1—N3	−1.2 (2)	C8—C9—C10—C5	−0.1 (3)
S1—N1—C1—N2	179.50 (11)	C6—C5—C10—C9	−0.5 (3)
C1—N2—C2—C3	0.4 (2)	S1—C5—C10—C9	175.75 (13)
N2—C2—C3—C4	−1.6 (2)	C15—N5—C11—N6	−178.70 (18)
C1—N3—C4—C3	2.0 (2)	C15—N5—C11—C12	0.1 (3)
C2—C3—C4—N3	0.3 (3)	N6—C11—C12—C13	177.36 (19)
O2—S1—C5—C10	24.88 (16)	N5—C11—C12—C13	−1.4 (3)
O1—S1—C5—C10	147.91 (14)	C11—C12—C13—C14	1.9 (3)
N1—S1—C5—C10	−99.39 (15)	C12—C13—C14—C15	−1.2 (3)
O2—S1—C5—C6	−158.80 (13)	C13—C14—C15—N5	0.0 (3)
O1—S1—C5—C6	−35.78 (16)	C11—N5—C15—C14	0.6 (3)
N1—S1—C5—C6	76.93 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1*N*···O2^{i}	0.86 (2)	2.04 (2)	2.895 (2)	168 (2)
N4—H2*N*···O1^{ii}	0.87 (2)	2.03 (2)	2.887 (2)	174 (2)
N5—H3*N*···N2^{iii}	0.92 (2)	1.85 (2)	2.758 (2)	174 (2)
N6—H4*N*···N4^{iv}	0.87 (3)	2.39 (3)	3.085 (2)	137 (2)
N6—H5*N*···N1^{iii}	0.92 (3)	2.00 (3)	2.918 (2)	173 (3)

Symmetry codes: (i) *x*−1, −*y*+3/2, *z*−1/2; (ii) *x*−1, *y*, *z*; (iii) −*x*+2, −*y*+2, −*z*; (iv) −*x*+1, −*y*+2, −*z*.

(VI)

Crystal data

$C_{12}H_{19}N_5O_4S$
M_r = 329.38
Monoclinic, $P2_1/c$
a = 12.7755 (4) Å
b = 9.8979 (4) Å
c = 11.5366 (4) Å
β = 93.508 (3)°
V = 1456.08 (9) Å^3
Z = 4

F(000) = 696
D_x = 1.503 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3829 reflections
θ = 3.2–29.5°
μ = 0.25 mm^{-1}
T = 123 K
Fragment, colourless
0.25 × 0.24 × 0.12 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: sealed tube
ω scans

Absorption correction: multi-scan
CrysAlis PRO, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40)
Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min} = 0.904, T_{max} = 1.000

7213 measured reflections
3571 independent reflections
2942 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 29.5°, θ_{min} = 3.2°
h = −16→17
k = −12→12
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.095
S = 1.06
3571 reflections
231 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.609P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.22358 (3)	0.19320 (4)	0.61905 (3)	0.01162 (11)
O1	0.17136 (8)	0.31689 (11)	0.58001 (10)	0.0164 (2)
O2	0.20716 (8)	0.15689 (11)	0.73914 (9)	0.0150 (2)
O3	0.45324 (9)	−0.36122 (12)	0.56317 (11)	0.0195 (3)
O1W	0.04450 (10)	0.06888 (14)	0.88727 (13)	0.0264 (3)
N1	0.34262 (10)	0.21715 (13)	0.59377 (11)	0.0131 (3)
N2	0.51428 (10)	0.15350 (13)	0.58746 (11)	0.0146 (3)
N3	0.39549 (10)	0.00832 (13)	0.68028 (11)	0.0150 (3)
N4	0.06716 (12)	−0.25029 (16)	0.31048 (14)	0.0204 (3)
N5	0.33848 (11)	−0.55735 (15)	0.42864 (13)	0.0153 (3)
C1	0.41755 (11)	0.12192 (16)	0.62242 (13)	0.0125 (3)
C2	0.59122 (12)	0.06494 (17)	0.61312 (14)	0.0167 (3)
H2	0.6595	0.0845	0.5894	0.020*
C3	0.57580 (13)	−0.05364 (17)	0.67255 (14)	0.0178 (3)
H3	0.6312	−0.1156	0.6906	0.021*
C4	0.47525 (12)	−0.07671 (17)	0.70408 (14)	0.0169 (3)
H4	0.4617	−0.1575	0.7450	0.020*
C5	0.17356 (11)	0.06143 (15)	0.53090 (13)	0.0119 (3)
C6	0.14954 (12)	−0.06369 (16)	0.57665 (14)	0.0149 (3)
H6	0.1589	−0.0789	0.6579	0.018*
C7	0.11201 (12)	−0.16636 (17)	0.50398 (14)	0.0163 (3)
H7	0.0946	−0.2513	0.5359	0.020*
C8	0.09943 (11)	−0.14617 (16)	0.38370 (14)	0.0150 (3)

C9	0.12428 (12)	−0.01923 (16)	0.33835 (13)	0.0150 (3)
H9	0.1163	−0.0040	0.2570	0.018*
C10	0.16023 (11)	0.08336 (16)	0.41125 (13)	0.0141 (3)
H10	0.1760	0.1693	0.3800	0.017*
C11	0.34327 (12)	−0.37296 (17)	0.57033 (15)	0.0184 (3)
H11A	0.3136	−0.2833	0.5878	0.022*
H11B	0.3284	−0.4349	0.6347	0.022*
C12	0.29135 (12)	−0.42582 (16)	0.45860 (15)	0.0173 (3)
H12A	0.2153	−0.4374	0.4673	0.021*
H12B	0.3005	−0.3601	0.3953	0.021*
H1N	0.0376 (17)	−0.223 (2)	0.243 (2)	0.033 (6)*
H2N	0.0370 (17)	−0.313 (2)	0.3425 (19)	0.029 (6)*
H3N	0.4051 (17)	−0.550 (2)	0.4209 (17)	0.023 (5)*
H4N	0.3120 (17)	−0.587 (2)	0.360 (2)	0.031 (6)*
H5N	0.3293 (17)	−0.625 (2)	0.483 (2)	0.032 (6)*
H1H	0.4656 (19)	−0.297 (3)	0.518 (2)	0.043 (7)*
H1W	0.095 (2)	0.097 (3)	0.833 (3)	0.065 (9)*
H2W	0.070 (2)	0.106 (3)	0.950 (2)	0.046 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01262 (17)	0.01188 (19)	0.01020 (19)	0.00106 (13)	−0.00062 (13)	−0.00008 (14)
O1	0.0180 (5)	0.0138 (6)	0.0170 (6)	0.0038 (4)	−0.0017 (4)	0.0010 (5)
O2	0.0167 (5)	0.0185 (6)	0.0099 (5)	0.0023 (4)	0.0007 (4)	0.0001 (4)
O3	0.0162 (5)	0.0161 (6)	0.0254 (7)	−0.0015 (5)	−0.0037 (5)	0.0028 (5)
O1W	0.0229 (6)	0.0330 (8)	0.0227 (7)	−0.0010 (5)	−0.0029 (5)	−0.0036 (6)
N1	0.0127 (6)	0.0129 (6)	0.0135 (6)	0.0000 (5)	−0.0011 (5)	0.0009 (5)
N2	0.0147 (6)	0.0149 (7)	0.0139 (6)	−0.0009 (5)	−0.0006 (5)	−0.0016 (5)
N3	0.0179 (6)	0.0144 (7)	0.0126 (6)	0.0016 (5)	0.0012 (5)	0.0008 (5)
N4	0.0253 (7)	0.0185 (8)	0.0171 (8)	−0.0046 (6)	−0.0014 (6)	−0.0029 (6)
N5	0.0164 (7)	0.0161 (7)	0.0131 (7)	−0.0015 (5)	−0.0010 (5)	0.0014 (6)
C1	0.0149 (7)	0.0137 (7)	0.0085 (7)	−0.0005 (6)	−0.0017 (5)	−0.0032 (6)
C2	0.0135 (7)	0.0206 (8)	0.0158 (8)	0.0009 (6)	0.0002 (6)	−0.0049 (7)
C3	0.0199 (8)	0.0184 (8)	0.0148 (8)	0.0061 (6)	−0.0016 (6)	−0.0017 (6)
C4	0.0222 (8)	0.0147 (8)	0.0136 (8)	0.0026 (6)	−0.0004 (6)	−0.0002 (6)
C5	0.0096 (6)	0.0138 (7)	0.0122 (7)	0.0000 (5)	0.0000 (5)	−0.0008 (6)
C6	0.0149 (7)	0.0188 (8)	0.0109 (7)	0.0001 (6)	0.0013 (5)	0.0010 (6)
C7	0.0172 (7)	0.0155 (8)	0.0161 (8)	−0.0031 (6)	0.0017 (6)	0.0012 (6)
C8	0.0111 (7)	0.0170 (8)	0.0169 (8)	0.0007 (6)	0.0007 (6)	−0.0036 (6)
C9	0.0152 (7)	0.0194 (8)	0.0104 (7)	0.0022 (6)	0.0003 (6)	0.0003 (6)
C10	0.0135 (7)	0.0154 (7)	0.0134 (7)	0.0008 (6)	0.0007 (5)	0.0026 (6)
C11	0.0171 (7)	0.0202 (8)	0.0178 (8)	−0.0006 (6)	0.0000 (6)	−0.0022 (7)
C12	0.0168 (7)	0.0159 (8)	0.0187 (8)	−0.0007 (6)	−0.0021 (6)	−0.0002 (7)

Geometric parameters (Å, º)

S1—O1	1.4525 (11)	C2—C3	1.379 (2)
S1—O2	1.4588 (11)	C2—H2	0.9500
S1—N1	1.5838 (13)	C3—C4	1.376 (2)
S1—C5	1.7505 (15)	C3—H3	0.9500
O3—C11	1.4172 (19)	C4—H4	0.9500
O3—H1H	0.84 (3)	C5—C6	1.388 (2)
O1W—H1W	0.97 (3)	C5—C10	1.397 (2)
O1W—H2W	0.86 (3)	C6—C7	1.384 (2)
N1—C1	1.3694 (19)	C6—H6	0.9500
N2—C2	1.337 (2)	C7—C8	1.401 (2)
N2—C1	1.3596 (19)	C7—H7	0.9500
N3—C4	1.337 (2)	C8—C9	1.405 (2)
N3—C1	1.346 (2)	C9—C10	1.379 (2)
N4—C8	1.379 (2)	C9—H9	0.9500
N4—H1N	0.89 (2)	C10—H10	0.9500
N4—H2N	0.83 (2)	C11—C12	1.507 (2)
N5—C12	1.484 (2)	C11—H11A	0.9900
N5—H3N	0.86 (2)	C11—H11B	0.9900
N5—H4N	0.90 (2)	C12—H12A	0.9900
N5—H5N	0.93 (2)	C12—H12B	0.9900
O1—S1—O2	114.34 (7)	C3—C4—H4	118.2
O1—S1—N1	104.16 (7)	C6—C5—C10	119.86 (14)
O2—S1—N1	114.03 (7)	C6—C5—S1	121.69 (12)
O1—S1—C5	107.67 (7)	C10—C5—S1	118.44 (12)
O2—S1—C5	107.43 (7)	C7—C6—C5	120.08 (14)
N1—S1—C5	108.97 (7)	C7—C6—H6	120.0
C11—O3—H1H	108.9 (16)	C5—C6—H6	120.0
H1W—O1W—H2W	101 (2)	C6—C7—C8	120.63 (15)
C1—N1—S1	121.16 (11)	C6—C7—H7	119.7
C2—N2—C1	116.93 (14)	C8—C7—H7	119.7
C4—N3—C1	116.59 (13)	N4—C8—C7	120.77 (15)
C8—N4—H1N	113.6 (15)	N4—C8—C9	120.40 (15)
C8—N4—H2N	114.6 (15)	C7—C8—C9	118.78 (14)
H1N—N4—H2N	116 (2)	C10—C9—C8	120.39 (14)
C12—N5—H3N	111.5 (13)	C10—C9—H9	119.8
C12—N5—H4N	111.2 (14)	C8—C9—H9	119.8
H3N—N5—H4N	104.9 (19)	C9—C10—C5	120.26 (14)
C12—N5—H5N	114.0 (13)	C9—C10—H10	119.9
H3N—N5—H5N	107.1 (18)	C5—C10—H10	119.9
H4N—N5—H5N	107.5 (19)	O3—C11—C12	111.39 (13)
N3—C1—N2	124.11 (14)	O3—C11—H11A	109.4
N3—C1—N1	122.02 (13)	C12—C11—H11A	109.4
N2—C1—N1	113.87 (14)	O3—C11—H11B	109.4
N2—C2—C3	122.86 (15)	C12—C11—H11B	109.3
N2—C2—H2	118.6	H11A—C11—H11B	108.0

C3—C2—H2	118.6	N5—C12—C11	109.89 (13)
C4—C3—C2	115.90 (15)	N5—C12—H12A	109.7
C4—C3—H3	122.1	C11—C12—H12A	109.7
C2—C3—H3	122.1	N5—C12—H12B	109.7
N3—C4—C3	123.61 (15)	C11—C12—H12B	109.7
N3—C4—H4	118.2	H12A—C12—H12B	108.2
O1—S1—N1—C1	178.29 (12)	N1—S1—C5—C6	109.60 (13)
O2—S1—N1—C1	52.98 (14)	O1—S1—C5—C10	43.52 (13)
C5—S1—N1—C1	−67.02 (14)	O2—S1—C5—C10	167.11 (11)
C4—N3—C1—N2	0.3 (2)	N1—S1—C5—C10	−68.88 (13)
C4—N3—C1—N1	−179.55 (14)	C10—C5—C6—C7	−0.3 (2)
C2—N2—C1—N3	−0.1 (2)	S1—C5—C6—C7	−178.76 (12)
C2—N2—C1—N1	179.74 (13)	C5—C6—C7—C8	1.0 (2)
S1—N1—C1—N3	−4.7 (2)	C6—C7—C8—N4	176.66 (15)
S1—N1—C1—N2	175.48 (10)	C6—C7—C8—C9	−0.8 (2)
C1—N2—C2—C3	−0.2 (2)	N4—C8—C9—C10	−177.60 (14)
N2—C2—C3—C4	0.3 (2)	C7—C8—C9—C10	−0.1 (2)
C1—N3—C4—C3	−0.1 (2)	C8—C9—C10—C5	0.8 (2)
C2—C3—C4—N3	−0.1 (2)	C6—C5—C10—C9	−0.6 (2)
O1—S1—C5—C6	−138.00 (12)	S1—C5—C10—C9	177.88 (11)
O2—S1—C5—C6	−14.40 (14)	O3—C11—C12—N5	56.48 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1*N*···O1*W*^{i}	0.89 (2)	2.34 (2)	3.173 (2)	156 (2)
N4—H2*N*···O1*W*^{ii}	0.83 (2)	2.58 (2)	3.293 (2)	144.1 (19)
N5—H3*N*···O3^{iii}	0.86 (2)	2.01 (2)	2.7762 (18)	147.2 (18)
N5—H4*N*···O2^{ii}	0.90 (2)	1.99 (2)	2.8488 (18)	159 (2)
N5—H4*N*···N3^{ii}	0.90 (2)	2.51 (2)	3.038 (2)	118.1 (17)
N5—H5*N*···O1^{iv}	0.93 (2)	2.44 (2)	3.1006 (18)	128.4 (17)
N5—H5*N*···N1^{iv}	0.93 (2)	2.02 (2)	2.933 (2)	167.3 (19)
O3—H1*H*···N2^{v}	0.84 (3)	1.90 (3)	2.7399 (19)	177 (2)
O1*W*—H1*W*···O2	0.97 (3)	1.94 (3)	2.9039 (17)	174 (3)
O1*W*—H2*W*···O1^{vi}	0.86 (3)	2.07 (3)	2.8997 (18)	163 (2)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) *x*, −*y*−1/2, *z*−1/2; (iii) −*x*+1, −*y*−1, −*z*+1; (iv) *x*, *y*−1, *z*; (v) −*x*+1, −*y*, −*z*+1; (vi) *x*, −*y*+1/2, *z*+1/2.

other supporting information

Crystallographic Information File. uk3153.cif

Structure factors. uk3153Isup2.hkl

Structure factors. uk3153IIsup3.hkl

Structure factors. uk3153IIIsup4.hkl

Structure factors. uk3153IVsup5.hkl

Structure factors. uk3153Vsup6.hkl

Structure factors. uk3153VIsup7.hkl